

**Influence of Texture and Grain Size**  
**on the Plastic Anisotropy in a Wrought Mg Alloy:**  
**Synchrotron X-ray Diffraction**  
**and Visco-plastic Self-consistent Modeling**

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## **DEDICATION**

This Doctoral dissertation is dedicated to my parents who gave me greatest love and continuous encouragement during my PhD study. Further dedication is given to other family members and friends for their supports.

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## ABSTRACT

The combined effects of texture and grain size on the yielding and hardening behavior in a hot-rolled AZ31B Mg alloy were studied by using synchrotron x-ray diffraction and visco-plastic self-consistent (VPSC) simulation methods.

First, the influence of texture on Hall-Petch relationships (namely, critical resolved shear stresses and hardening parameters) in a Mg alloy was investigated to establish a constitutive basis for the VPSC simulation.

Then, the changes in dominant deformation mechanisms (basal, prismatic, and pyramidal slip as well as extension twin) and their relative interactions were studied systematically as a function of the initial texture using a VPSC scheme. The simulation results provide basic understanding of the effect of texture on the hardening behaviors in terms of the interaction of various active deformation mechanisms.

Subsequently, the interplay between the texture and grain size effects on the activation of multiple deformation mechanisms and the plastic anisotropy was studied by incorporating the Hall-Petch Relationship in the VPSC modeling scheme.

Finally, the influence of  $\{10\bar{1}2\}$  extension twin on the texture and microstructure evolution was investigated to understand physical implications of the simulation results. Specifically, the hardening anisotropy introduced by extension twin was studied with a focus on dislocation-twin interactions in terms of texture hardening, size hardening, and glissile-to-sessile transition hardening.

The current study provides a basic micromechanical understanding of the effects of complex interactions among texture, loading path, and grain size on the yielding and hardening behavior of a wrought Mg alloy through plasticity simulations and diffraction measurements. The results would help improving the formability and advancing manufacturing techniques of wrought Mg alloy components for structural applications.

Keywords: Magnesium, Grain size, Texture, Slip, Twin

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## **Chapter 1**

### **Introduction and General Information**

The light weight and high specific strength are the main driving forces behind the development and application of Mg alloys as a structural material in transportation and 3C (computer, communication, and consumer electronic) industries. The density of pure magnesium is  $1.74 \text{ g/cm}^3$ , while that of aluminum is  $2.70 \text{ g/cm}^3$ , and steel  $7.85 \text{ g/cm}^3$ . The specific strength of Mg alloys are higher than most of the structural materials. Only some advanced aluminum alloys such as 8000 series and advanced stainless steels such as martensitic steels have the comparable specific strength as Mg alloys. It is reported that [1], up to 26 kg of magnesium alloys are used in the GM full-size Savana & Express vans. These vehicles use cast AZ91D magnesium transmission casings, offering 20%-25% weight savings over aluminum.

However, the growth in the application of Mg alloys is mainly in the area of die-cast components. The relatively poor workability and low ductility inhibit the broad application of wrought Mg products despite their advantages [1]. Moreover, the high anisotropy during the deformation due to the strong influence of texture on the activation of different deformation mechanisms increases the complexity in shaping and using the Mg alloys. This is a common issue for the alloys with low crystal symmetry including the hexagonal close-packed (HCP) Mg alloys.

Thermo-mechanical processes; such as equal channel angular pressing (ECAP), friction stir processing (FSP), hot extrusion, and hot rolling; change the texture and grain size of most alloys simultaneously and significantly. A distinct initial texture in Mg alloy, in particular, may cause activations of particular deformation mechanisms (basal, prismatic, or pyramidal slip as well as the extension twin). The activation of a particular deformation mechanism, in turn, leads to a distinct texture evolution. In particular, extension twin causes drastic changes in texture and also gives rise to complex interactions with slip and extension twin itself. As a result, a dramatic anisotropy in yielding and hardening behavior are observed for the Mg alloys at ambient temperature.

Moreover, the grain size affects the mechanical properties of Mg alloys following the Hall-Petch (H-P) relationship. In fact, a wide range of parameters of H-P relationship was reported in the literature for Mg alloys subjected to different processing methods. It indicates that the grain-size sensitivity, which is represented by the H-P parameters, may be highly dependent on the texture of Mg alloys.

To date, the combined effects of texture and grain size on the mechanical behavior of wrought Mg alloys have not been studied in depth. Therefore, the main goal of the current study is to gain fundamental understanding of the interplay between the texture and grain size effects on the plasticity of Mg alloys using synchrotron x-ray diffraction (S-XRD) measurements and visco-plastic self-consistent (VPSC) model simulations. Specific goals and scientific issues of this study are:



- To gain fundamental understanding of the interaction between the texture and deformation mechanisms in Mg alloys:
  - effects of initial texture on the activation of different deformation mechanisms
  - effects of a particular deformation mode on the subsequent texture evolution
  - effects of the activation of various deformation modes and their interactions on the hardening behavior
- To understand the grain size effect on the yielding and hardening behavior of Mg alloys with different initial texture
  - relationship between initial texture and parameters of H-P relationship ( $\sigma_0$  and  $k_y$ )
  - effects of grain size on the activations of different deformation mechanisms and hardening behavior
- To determine the effect of extension twinning on the hardening behavior
  - influence of loading path (e.g., *c*-tension vs. *a*-compression) on the twin variant selection
  - effects of extension twin variant selection on texture evolution, grain size (mean free path for dislocations), twin-slip interactions, and hardening behavior

The current dissertation consists of five main parts. A comprehensive literature review (**Chapter 2**) is provided on (1) general review of Mg alloy types, properties, and applications, (2) plastic deformation (i.e., slip and twin) mechanisms of Mg alloys, (3) texture effects, (4) grain-size effects, (5) VPSC model theories, current development

efforts, and examples, and (6) application of diffraction for deformation micromechanics studies.

Subsequent four chapters provide experimental and simulation results and related discussions on four sequential research topics as follows.

The “Influence of Texture on Hall-Petch Relationships in a Mg Alloy” is present in **Chapter 3**. This study establishes a constitutive basis for the VPSC simulation. Specifically, critical resolved shear stresses and hardening parameters for various deformation mechanisms active in Mg are experimentally measured and also predicted using dislocation pile-up theory.

The “Interplay between Texture, Loading Path, and Active Deformation Mechanism in a Wrought Mg Alloy” is presented in **Chapter 4**. The changes in dominant deformation mechanisms (namely, basal, prismatic, and pyramidal slip as well as extension twin) and their relative interactions are studied systematically as a function of the initial texture using a VPSC scheme. The simulation results provide basic understanding of the effect of texture on the flow and hardening behaviors in terms of the interaction of various active deformation mechanisms.

In **Chapter 5**, “Visco-Plastic Self-Consistent Modeling of the Hall-Petch Relationships for Various Deformation Mechanisms in a Mg Alloy” is presented. In this chapter, our effort on incorporating the Hall-Petch Relationship in the VPSC modeling scheme is reported. The simulation results show an excellent agreement with the experimental results. The interplay between the texture and grain size effects on the activation of multiple deformation mechanisms and the plastic anisotropy is discussed.

Finally, “Role of  $\{10\bar{1}2\}$  Extension Twin in the Hardening Behavior of a Mg Alloy” is presented in **Chapter 6**. The  $\{10\bar{1}2\}$  twin variant selection, microstructure, texture, and active deformation mechanisms were investigated to understand hardening mechanisms. Specifically, the hardening anisotropy introduced by extension twin was studied with a focus on dislocation-twin interactions in terms of texture hardening, size hardening, and glissile-to-sessile transition hardening.

## **Chapter 2**

### **Literature Review**

#### **2.1 General introduction of Mg alloys**

##### ***2.1.1 Various types of Mg alloys***

Magnesium is the third most commonly used structural metal, following iron and aluminum. Magnesium alloys are mixture of magnesium with other metals like aluminum, zinc, manganese, zirconium, silicon, copper and rare earths. Main commercial magnesium alloys include the AZ series (Mg-Al-Zn), AM series (Mg-Al-Mn), AE series (Mg-Al-RE), EZ series (Mg-RE-Zn), ZK series (Mg-Zn-Zr), and WE series (Mg-RE-Zr). Magnesium alloys can be divided into cast and wrought magnesium alloys in terms of the processing condition.

Overall, more than 90% of the magnesium alloys used as structural components are produced by casting process, especially by die-casting processes. In the last decade, the AZ series (AZ91) and AM series alloys (AM50A, and AM60B) cast magnesium alloys have been extensively studied and used for some structural components of automobiles, aircraft, and computers. These alloys have good mechanical properties, corrosion resistance, and die-castability. However, the poor creep resistance above 125°C inhibits the wide application of these alloys in the major powertrain, since the automatic transmission is under serve at 175 °C, engine blocks up to 200 °C, and engine pistons at 300 °C.

Actually, wrought magnesium alloys have a more promising perspective of application because of the improved mechanical properties compared with cast magnesium alloys. However, the low ductility and poor workability set up the barriers of broader application of wrought Mg and Mg alloys even though they have better performance than cast magnetism. Therefore, the tremendous interesting is attracted for the improvement of the formability of the wrought Mg alloy in the academic research field, which may cause significant impact for the application of magnesium in industry.

A long time research plan of wrought magnesium alloys was put forward by the international magnesium association (IMA) in 2000 to study the plastic deformation, microstructure and mechanical behavior of wrought Mg alloys. Moreover, the superplasticity of the wrought AZ31, AZ61, and ZK60 have been achieved after the severe plastic deformation introduced by friction stir processing (FSP) and equal channel angular pressing (ECAP) [2-4]. Recently, some rare earth elements such as Gd and/or Y, Nd, Dy are added in the wrought magnesium alloys to obtain the higher specific strength at both room and elevated temperatures, especially, the improved creep resistance as the noble Mg alloys. The alloys additions in wrought magnesium alloys can be divided into three categories: (1) elements that can improve both strength and ductility of magnesium including Al, Zn, Ca, Ag, Ce, Ni, Cu, Th; (2) elements that can only improve ductility, but with a little effect on strength of magnesium such as Cd, Ti, and Li; and (3) elements that decrease the ductility but increase strength of magnesium including Sn, Pb, Bi, and Sb.[5]

### ***2.1.2 Typical physical, mechanical, and corrosion properties of wrought Mg alloys***

The typical physical properties of the common commercial Mg alloys are listed in Table 2-1. The melting temperature of Mg alloys is about 600°C.

The mechanical properties of wrought Mg alloys are dependent on the compositions and forming process. The ductility of Mg alloys at low temperature is not as good as face-centered cubic (FCC) alloys such as aluminum and copper, which gives the trouble of cold working for Mg alloys. The strength decrease and plasticity increase obviously with the increase in temperature so that the formability is improved during the thermal precessing. The mechanical properties are related to deformation mechanisms in HCP alloys, which are highly dependent on the texture. The understanding of the relationship between the mechanical properties and deformation mechanisms can help us to improve the cold-formability and mechanical properties of Mg alloys, which will be the main part of the following review and the motivation of my thesis research.

For magnesium and its commercial alloys, the modulus of elasticity in tension and compression is about 45GPa. The modulus of elasticity in shear, or modulus of rigidity, is about 16GPa. Poisson's ratio is 0.35. Due to the asymmetry of HCP structure, Mg single crystal has strong anisotropy of deformation. The mechanical properties of wrought Mg alloys are determined by the composition. For AZ31B, whose composition is listed in Table 2-2, the typical tensile strength is 260MPa, the yield strength is 175MPa and the ductility is around 15% at ambient temperature [6].

Table 2-1 Typical physical properties of Mg alloys [7]

| Property                                | Unit    | Temp<br>(F) | AZ91    | AM60    | AM50    | AM20    | AS41    | AS21    | AE42 |
|-----------------------------------------|---------|-------------|---------|---------|---------|---------|---------|---------|------|
| Density                                 | g/cu cm | 68          | 1.81    | 1.8     | 1.77    | 1.75    | 1.77    | 1.76    | 1.79 |
| Liquidus Temperature                    | F       |             | 1110    | 1139    | 1148    | 1182    | 1144    | 1169    | 1157 |
| Incipient Melting<br>Temperature        | F       |             | 788-815 | 788-815 | 788-815 | 788-815 | 788-815 | 788-815 | 1094 |
| Linear Thermal<br>Expansion Coefficient | Mm/m    | 68-212      | 26      | 26      | 26      | 26      | 26.1    | 26.1    | 26.1 |
| Specific Heat of Fusion                 | kJ/kg   |             | 370     | 370     | 370     | 370     | 370     | 370     | 370  |
| Specific Heat                           | kJ/kg*K | 68          | 1.02    | 1.02    | 1.02    | 1.02    | 1.02    | 1.02    | 1.02 |
| Thermal Conductivity                    | W/K*m   | 68          | 51      | 61      | 65      | 94      | 68      | 84      | 84   |
| Electrical Conductivity                 | MS/m    | 68          | 6.6     | nm      | 9.1     | 13.1    | nm      | 10.8    | 11.7 |

Table 2-2 The composition of AZ31

| Elements | Al      | Zn      | Mn      | Si    | Cu     | Ni     | Mg   |
|----------|---------|---------|---------|-------|--------|--------|------|
| wt. %    | 2.5-3.5 | 0.6-1.4 | 0.2-0.6 | <0.05 | <0.008 | <0.002 | Bal. |



The mechanical properties are also dependent on the operation temperature and strain rate, which determines the creep properties of Mg alloys. Overall, the ductility increases with the increase in temperature or decrease in the strain rate, while the strength is in the opposite trend.

Magnesium alloys have a poor corrosion resistance, since the high electron negative potential, especially in the salty environment. The oxide layer can be formed at surface to protect the oxidation to some extent. However, the Cl ions can penetrate the oxide layer reaching the surface of the alloy then reacting with the metal substrate relatively easy, since the oxide layer is non-compact. Moreover, impurities and second phases act as active cathodic sites causing local galvanic acceleration of corrosion of the matrix. The corrosion performance of magnesium alloys in sodium chloride solutions is affected by other factors such as alloying elements, precipitations, microstructure, and grain size. The alloys containing low amounts of iron, copper and nickel are more corrosion resistant.

The coatings and surface treatments used in industry to protect magnesium alloys are, oils and waxes for temporary protection; chemical-conversion coatings for temporary protection or paint base; anodized coatings for wear resistance as well as a superior paint base; paints and powder coatings used for corrosion protection and appearance; metallic plating is good for appearance, surface conductivity, solderability and limited corrosion protection; and adding corrosion inhibitors for increasing the surface resistance by isolating from being in contact with the corrosive environment.[8]

### ***2.1.3 Traditional and emerging applications of Mg alloys***

Magnesium's attractive properties such as low density, high specific stiffness and strength, good heat conduction and electro-magnetic interference shielding effects determine the wide variety of application of Mg alloys.

The most important areas for the application of magnesium and magnesium alloys include the automotive, aerospace and electronic devices [9]. The strong and light characters make magnesium widely used in several high volume part manufacturing applications such as automotive and truck components. Specialty, high-grade car wheels of magnesium alloys which are called 'mag wheels' have been used [10]. Magnesium alloys are also used for the body parts of cars especially the racing cars in order to reduce the weight of the cars as early as 1950s. The Mercedes-Benz race car model, the Mercedes-Benz 300 SLR, had a body made from Elektron, a magnesium alloy and won a lot of world-class race events in 1955. Some others famous brands of automotive manufactories including Chevrolet, Porsche, BMW also use magnesium alloys to get higher performance. Recently, the use of magnesium alloys is even more widely. The high temperature low creep magnesium alloys such as AJ62 are used for engine blocks, as featured in the new BMW 325i and 330i models [11]. The alloy development and reduce of the costs can broaden the application of Mg in the automotive filed by competing with the Al alloys.

Historically, magnesium was one of the main aerospace construction metals and was used for German military aircraft as early as World War I and extensively for German aircraft in World War II. The application of magnesium in the commercial

aerospace industry was generally restricted to engine related components because of the perceived hazards with magnesium parts in the event of fire. Currently the use of magnesium alloys in aerospace is increasing, mostly driven by the increasing importance of fuel economy and the need to reduce weight. The development and testing of new magnesium alloys continues, notably Elektron 21, which has successfully undergone extensive aerospace testing for suitability in engine internal and airframe components.

The third application field of magnesium is electronic devices. Magnesium is widely used for manufacturing of mobile phones, laptop computers, cameras, and other electronic components because of low weight, as well as good mechanical and electrical properties,.

The cast magnesium alloys are the most used Mg alloys, which can be produced by nearly all of the conventional casting methods, which include sand, permanent, and semi-permanent mold and shell, investment, and die-casting [12]. The choice of a casting method for a particular part depends upon factors such as the configuration of the proposed design, the application, the properties required, and the total number of castings required.

Some wrought magnesium alloy includes AZ31, AZ61, ZK60, HK31 are used in the industry as well [13]. Wrought magnesium alloy have special features. After forming, wrought magnesium alloys have a stringy texture in the deformation direction which causes the anisotropic deformation for compression and tension. The most important application of wrought magnesium alloy are photoengraving plate made of AZ31 plate, sacrificial anode used to protect steel heat exchangers made of extruded magnesium

alloy. However, the application of wrought magnesium alloy is inhibited because of its low formability. Therefore, there has been a great deal of interest in developing a better understanding of the deformation of magnesium to improve the deformation-processing technologies for magnesium alloys. Considering the better mechanical properties of wrought alloy than cast one, the improvement of formability of magnesium alloy will have a widespread application in the near future.

#### ***2.1.4 Main advantages of Mg alloys for structural application***

Magnesium alloys have always been attractive to designers due to their low density ( $1.74\text{g/cm}^3$ ), which is 34% lighter than aluminum ( $2.70\text{g/cm}^3$ ) and 76% lighter than steel ( $7.8\text{g/cm}^3$ ) and about the same as many plastics. The products made by magnesium can reduce the weight obviously. It is reported that car manufacturers already plan to boost magnesium content in automobiles to between 45-160kg, up from just about 5kg in the typical car today and lead to reducing weight by about 22.5kg than before. It has been estimated that it could increase the vehicle fuel efficiency by around 1%, which means the vehicles could save 100,000 barrels of oil per day in nationwide [14].

Meanwhile, the magnesium alloys have high strength and stiffness. The Young's modulus of AZ91D is 20 times stiffer than typical engineering thermoplastics, and the ultimate tensile strength (UTS) is about 240MPa which shows high strength to weight ratio [6]. The comparison of specific strength (UTS/density) and specific stiffness between magnesium alloys, aluminum alloys, low carbon steel, stainless steel and some common plastics are shown in Figure 2-1 and Figure 2-2.

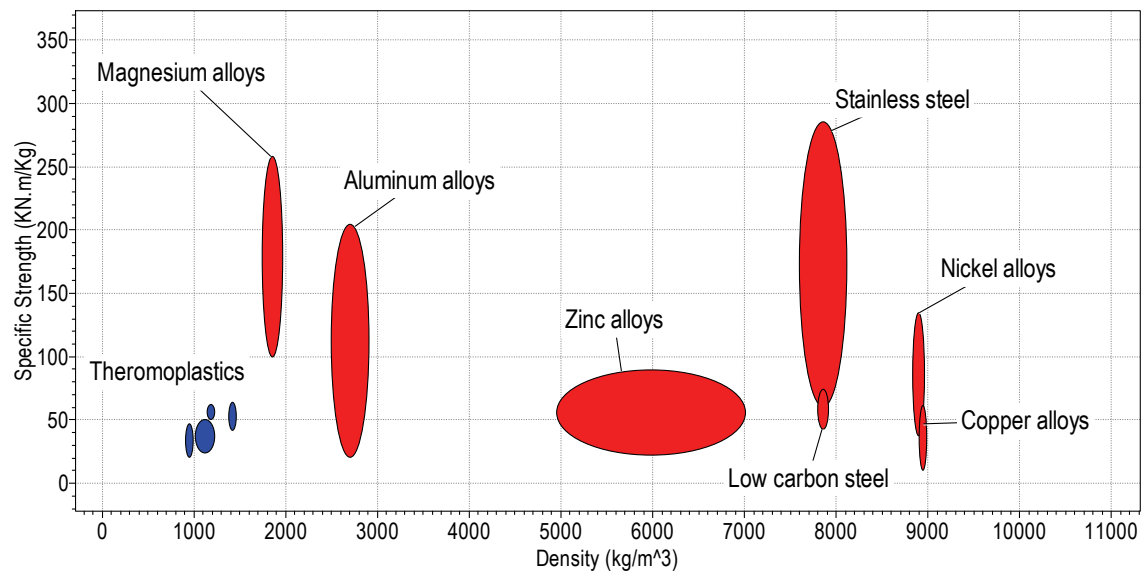


Figure 2-1 Comparison of specific strength of magnesium alloys with other alloys and thermoplastics.

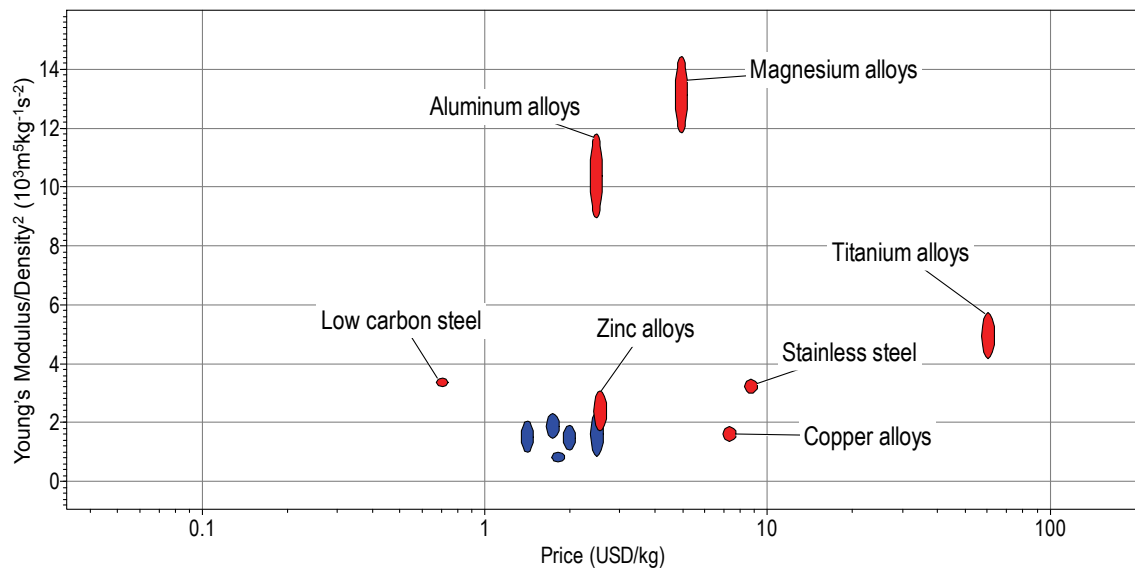


Figure 2-2 Comparison of specific stiffness of magnesium alloys with other alloys and thermoplastics.

It is clear shown that the magnesium alloys have low density compare to other alloys and higher specific stiffness and specific strength. The specific strength of Mg alloys is higher than both thermoplastics and the most widely used structural metals such as low carbon steels, copper alloys, zinc alloys, nickel base superalloys and most aluminum alloys. Only the most advanced aluminum alloys such as 8000 series and some advanced steels such as TRIP steels, duplex steels shows the similar specific strengths as that of Mg alloys.

Moreover, the magnesium alloys also have higher vibration absorption. At a stress of 35MPa, attenuation coefficient is 25% for AZ91D while only 1% for A380 [15]. Magnesium alloys have good machinability. The effective EMI shielding and high thermal management are also advantages of magnesium alloys [16]. Magnesium alloys are also fully recyclable which make them environmental friendly. Magnesium is one the earth's most abundant elements, which make the magnesium alloys are also cost-effective.

#### ***2.1.5 Key drawbacks of Mg for structural application***

Although there are a lot of benefits for the use of magnesium alloys, some drawbacks for the Mg application inhabit the development. First of all, magnesium metal and its alloy are explosive hazards. They are highly flammable in their pure form when molten or in powder or in ribbon form. Secondly, magnesium alloys have high tendency to galvanic corrosion when they contact with dissimilar metals or electrolyte [17]. The more severe issue is that it is hard to deform by cold working, which increase the cost for

deforming Mg alloys. Thus, more researches were done to overcome the disadvantages of Mg, especially the wrought magnesium alloys.

## **2.2 Plastic deformation the of Mg alloys**

The slip systems and deformation twinning systems are two most important deformation mechanisms, which determine the plastic deformation behavior of Mg alloys. Lots of researchers [18-23] have studied dislocation theory for Mg alloys, which includes characteristics of dislocation slip, the cross slip of dislocation, the interaction of the dislocations to dislocations, dislocation to grain boundaries and dislocation to twins. The twinning mechanisms in the deformation of Mg alloys are also analyzed including the effect of twin on the texture evolution, hardening rate, and twin-twin interaction. The factors such as strain rate, temperature, grain size, composition of alloys which can affect the deformation mechanisms and mechanical properties are evaluated to clarify the plasticity theory of Mg alloys.

### **2.2.1 Introduction of the crystallography of Mg alloys**

The space group of Mg is  $P63/mmc$ , which is HCP structure. The atomic packing factor of HCP is as high as 0.74, which is equal to the face centered cubic (FCC) structure, since in both structures the atoms are packed as tightly as possible, and the total number of atoms in the unit cell is 6. In both HCP and FCC structure each atom is surrounded by 12 other atoms so that the coordination number is 12. However, their crystal structures are not identical because the stacking arrangements of the atomic plane for FCC and HCP



structures are different. The  $\{111\}$  planes of the FCC crystal structure have the identical packing arrangement as the  $\{0001\}$  planes of the HCP crystal structure which are both the close-packed plane for each structure. The stacking arrangement for HCP is ABABABA... which is distinct to that ABCABCA... arrangement of FCC structures.

The symmetry of HCP structure is much lower than that of the cubic system. The ratio of the height  $c$  of the hexagonal prism of the HCP crystal structure to its basal side  $a$  is called the  $c/a$  ratio. The  $c/a$  ratio for an ideal HCP crystal structure consisting of uniform spheres packed as tightly together as possible is 1.633. The easiest slip plane of HCP structure is determined on the  $c/a$  ratio. When  $c/a$  ratio is bigger than 1.633, such as the pure Mg and most Mg alloys, which indicates that the atoms in these structures are slightly elongated along the  $c$ -axis of HCP unit cell, the most close-packed plane is the  $\{0001\}$  basal plane, which makes it as the easiest slip plane. On the other hand, for the structures which has the smaller  $c/a$  ratio ( $<1.633$ ) like zinc ( $c/a=1.589$ ) and titanium ( $c/a=1.587$ ), the distance between two basal slip decreases because of the slightly compress along the  $c$ -axis. Therefore, the most close-packed plane is  $(10\bar{1}0)$  prismatic slip plane or  $(11\bar{2}2)$  pyramidal plane [24].

### **2.2.2 Slip in Mg alloys**

The characteristics of dislocations in Mg alloys are different from those in copper, aluminum and steels because of the low symmetry of HCP structures. The dislocations in Mg alloys includes  $\langle a \rangle$  dislocation and  $\langle c+a \rangle$  dislocation [25]. These dislocations can slip along various planes such as basal, prismatic, and pyramidal plane.

### 2.2.2.1 The dislocations in Mg alloys

The main dislocations in Mg alloys includes  $\langle a \rangle$  dislocation,  $\langle c+a \rangle$  dislocation, and some partial dislocations. The burgers vector of  $\langle a \rangle$  dislocation is  $a/3 \langle 11\bar{2}0 \rangle$ , which is the smallest one in Mg alloys. Therefore,  $\langle a \rangle$  dislocation has the strongest mobility, which can move along basal, prismatic and pyramidal plane.

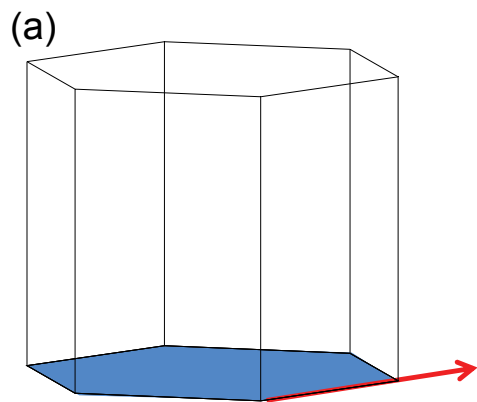
Another common dislocation in Mg alloy is  $\langle c+a \rangle$  dislocation, whose burgers vector is  $\sqrt{c^2 + a^2} \langle 11\bar{2}3 \rangle$ . Comparison to  $\langle a \rangle$  dislocation,  $\langle c+a \rangle$  dislocation has much larger Burgers vector and smaller plane distance. Therefore, the mobility of  $\langle c+a \rangle$  dislocation is smaller. S. Ando [18] reported that  $\langle c+a \rangle$  edge dislocations have two types of core structures, one is a perfect dislocation, which is extend to basal plane and cannot move. When the core structure of  $\langle c+a \rangle$  edge dislocation becomes a core with small extension core on the basal plane, the core changes to another type, which is consist of two partial dislocations and it can easily move on  $(11\bar{2}2)$  slip plane by applying stress.  $\langle c+a \rangle$  dislocation can form through the interaction of  $\langle a \rangle$  and  $\langle c \rangle$  dislocation;  $\langle a \rangle$  dislocation and  $\{11\bar{2}2\}$  twin; or  $\langle c \rangle$  dislocation and  $\{11\bar{2}1\}$  twin.

Some partial dislocation can also exist in HCP structure [26]. The  $\langle a \rangle$  dislocation is easy to dissociate to two Shockley partial dislocations on basal plane due to the low stacking fault.  $\frac{a}{3} \langle 11\bar{2}0 \rangle = \frac{a}{3} \langle 10\bar{1}0 \rangle + \frac{a}{3} \langle 01\bar{1}0 \rangle$ . On the other hand,  $\langle a \rangle$  dislocation can also dissociate on prismatic plane as  $\frac{a}{3} \langle 11\bar{2}0 \rangle = \frac{a}{6} \langle 11\bar{2}x \rangle + \frac{a}{3} \langle 11\bar{2}\bar{x} \rangle$ , where  $x$  is range in 0~1 for most Mg alloys. The large distance of extended dislocation leads to the low mobility of dislocation [27]. Therefore, the cross-slip of dislocation in Mg alloys is not easy to occur at room temperature.

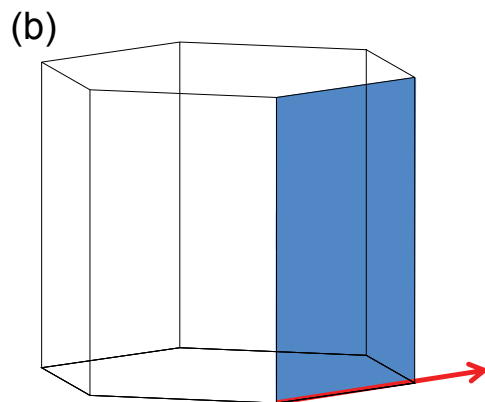
#### 2.2.2.2 The independent slip systems in Mg alloys

The atoms are close-packed on (0001) basal plane so that it is the basic slip system in Mg alloys. The slip direction is along  $\langle 11\bar{2}0 \rangle$ . In this case, two independent slip systems can be offered on basal plane (Figure 2-3a). Similarly,  $\langle 11\bar{2}0 \rangle$  dislocation can also slip on the prism plane (10 $\bar{1}$ 0) (Figure 2-3b). Thus, another two independent slip systems can also be provided. However, normally, prismatic slip is not easy to be activated because the critical resolved shear stress (CRSS) for prismatic slip is much larger than that for basal plane at room temperature [28]. The increase in temperature or decrease in the grain size to less than 10 $\mu\text{m}$  can improve the activity of prismatic slip system.

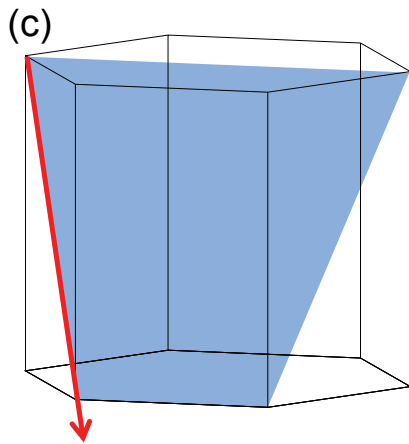
Pyramidal planes are another type of slip system in Mg alloys, especially at relatively high temperature.  $\langle a \rangle$  dislocation can slip on pyramidal plane, but it is crystallographically equivalent to the combined four independent modes by cross slip between basal and prismatic slip system. Therefore, when cross-slip is taking place, a total of only four independent modes are provided with  $\langle a \rangle$  vectors. On the other hand,  $\langle c+a \rangle$  dislocations whose Burgers vector is  $\langle 11\bar{2}3 \rangle$  can also slip along pyramidal planes. The possible pyramidal planes include {10 $\bar{1}$ 1}, {11 $\bar{2}$ 1}, {10 $\bar{1}$ 2} and {11 $\bar{2}$ 2}. The slip direction is always  $\langle 11\bar{2}3 \rangle$ . When a slip system with  $\langle c+a \rangle$  Burgers vectors is operative, this alone provides five independent modes, which is sufficient to fulfill von Mises criterion.



Basal  $\langle a \rangle : \{0001\} \langle 11\bar{2}0 \rangle$



Prismatic  $\langle a \rangle : \{10\bar{1}0\} \langle \bar{1}2\bar{1}0 \rangle$



Pyramidal  $\langle c+a \rangle : \{1011\} \langle 11\bar{2}3 \rangle$

Figure 2-3 Slip systems in Mg alloys: (a): basal slip, (b) prismatic slip, and (c) pyramidal slip

The  $\langle c+a \rangle$  dislocation is very important for the deformation and ductility of Mg alloys not only because of the sufficient independent slip systems to fulfill Von Mises criterion, but also because of the accommodation of strain and stress along  $c$ -axis of crystals. The  $\langle c+a \rangle$  pyramidal slip can inhibit the anisotropic deformation of Mg and increase the ductility and formability of Mg alloys. However, the  $\langle c+a \rangle$  slip on pyramidal plane associates with the largest Burgers vector, the smallest interplanar spacing, hence the narrowest dislocation width, and the hardest in gliding [29]. Thus, the CRSS for  $\langle c+a \rangle$  dislocation is always much larger than the basal and prismatic slip in Mg alloy at room temperature and the slip only happens at the region where high stress is concentrated such as at grain boundaries, twin boundaries and the conjunction of  $\langle a \rangle$  and  $\langle c \rangle$  dislocations [30].

Another issue is the stability of a  $\langle c+a \rangle$  dislocation against  $\langle a \rangle$  and  $\langle c \rangle$  dislocations.  $\langle c+a \rangle$  dislocations can be a pure edge dislocation, pure screw dislocation or mixed dislocation. Analysis of  $\langle c+a \rangle$  dislocation through transmission electron microscopy (TEM) demonstrates that  $\langle c+a \rangle$  dislocation are always found at the conjunction of  $\langle a \rangle$  and  $\langle c \rangle$  dislocations, which means that only screw  $\langle c+a \rangle$  dislocation is stable, while the edge dislocation is the least stable one [31]. However, the relation between the  $\langle c+a \rangle$  dislocation with  $\langle a \rangle$  and  $\langle c \rangle$  dislocation and the reaction between them are not quite clear right now.

### 2.2.3 Twinning in Mg alloys

It clearly shows that  $\langle a \rangle$  dislocation slip along basal, prismatic or pyramidal planes provide only a total of four independent slip systems and none of them is capable of accommodating any plastic strain along the crystallographic  $c$  direction. The activation of second pyramidal  $\langle c+a \rangle$  slip system is generally assumed to provide the additional degree of freedom needed for an arbitrary isochoric plastic strain, and has been confirmed experimentally [27]. On the other hand, deformation twinning can also provide the needed  $c$ -axis strain component for generalized deformation. Two common twin modes have been observed in magnesium and its alloys. These have classified as  $\{10\bar{1}2\} \langle \bar{1}011 \rangle$  extension twins (shown in Figure 2-4) and  $\{10\bar{1}1\} \langle 10\bar{1}2 \rangle$  contraction twins, because they results in the extension and the contraction of the crystal along the crystallographic  $c$  direction, respectively. The twin has the interaction with the dislocation at the twin boundaries and influent the plastic deformation of Mg alloys [32].

#### 2.2.3.1 Twinning systems in Mg alloys

Slip, twin and fracture are competition with each other to release strain in a wide range of temperature. The most common twin systems in Mg alloys include two sets:  $\{10\bar{1}2\}$  and  $\{10\bar{1}1\}$ .

$\{10\bar{1}2\}$  twin is the easiest twin systems to be activated due to the smallest shear strain in HCP structure. The twin elements and shear strain are [19]:

$$K_1 = \{10\bar{1}2\}; K_2 = \{10\bar{1}2\}; \eta_1 = \langle 10\bar{1}1 \rangle; \eta_2 = \langle 10\bar{1}1 \rangle; P = \{1\bar{2}10\};$$

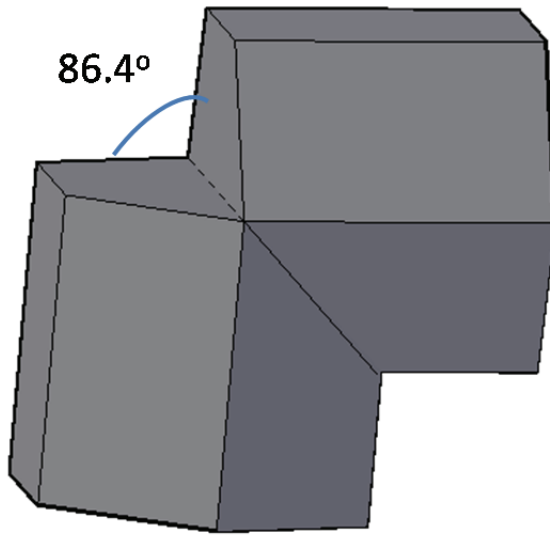
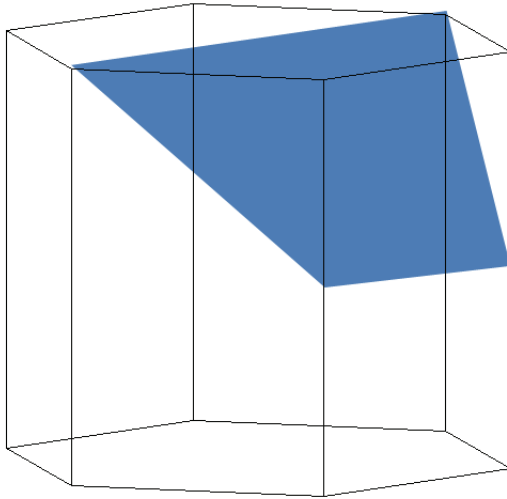


Figure 2-4  $\{10\bar{1}2\} <\bar{1}011>$  extension twinning for Mg alloy

$$s = \frac{\sqrt{3}(\gamma^2 - 3)}{3\gamma} = -0.13.$$

The extension twinning can only be activated when  $c$ -axis is stretched which means tension along  $c$ -axis or compression perpendicular to  $c$ -axis [33].

The other common twin system in Mg alloy is  $\{10\bar{1}1\}$  contract twin [34]. Two modes of  $\{10\bar{1}1\}$  contract twin are presented:  $q=4$  or  $q=8$ . For  $q=4$  mode, the twin elements is  $K_1 = \{10\bar{1}1\}$ ;  $\eta_2 = \langle 41\bar{5}3 \rangle$ ;  $K_2$  and  $\eta_1$  are irrational. Another mode:  $q=8$  was presented by R. Hill which has a lower shear strain. The twin element is:

$$K_1 = \{10\bar{1}1\}; K_2 = \{10\bar{1}\bar{3}\}; \eta_1 = \langle 10\bar{1}\bar{2} \rangle; \eta_2 = \langle 30\bar{3}2 \rangle; P = \{1\bar{2}10\};$$

$$s = \frac{4\gamma^2 - 9}{4\sqrt{3}\gamma} = 0.138$$

The contraction twin are generally activated at high temperature deformation ( $T > 673\text{K}$ ) [35]. Some other types of twinning such as  $\{30\bar{3}4\}$ ,  $\{10\bar{1}3\}$ , and  $\{11\bar{2}3\}$  can also be activated in some cases.  $\{30\bar{3}4\}$  twin can be looked as the double twin in Mg alloys which is the  $\{10\bar{1}2\}$  twin happened in  $\{10\bar{1}1\}$  twin.

### 2.2.3.2 The interaction between twinning and dislocation in Mg alloys

The interaction of twin with dislocations which have  $\langle a \rangle$ ,  $\langle c \rangle$  or  $\langle a+c \rangle$  Burgers vector has been studied. In Mg alloys, the prismatic or basal slip can be obstacle by the  $\{10\bar{1}2\}$  twin, which causes the local stress concentration around the twin boundaries by the dislocation pile-up [36]. The stress concentration may become a site for the nucleation of conjugate twins or formation of crack, depending on the cleavage strength of metal. If the fracture strength is much higher than the twin nucleation, the twin



interface may become the effective sites for nucleation of other twins. On the other hand, the crack can be initiated at or near the twin interface [27, 33, 37].

Moreover, twin interfaces can also be the sources of  $\langle c+a \rangle$  dislocations. The interaction of  $\langle a \rangle$  dislocations with  $\{10\bar{1}2\}$  twin can give rise to the  $\langle c+a \rangle$  dislocation and the partial dislocation on the twin interface by the reaction:  $\frac{a}{3}\langle\bar{2}110\rangle_B = \frac{a}{6}\langle11\bar{2}\bar{3}\rangle_{TP} - b_T$ . In this case, the interaction of twin with dislocation will improve the plasticity of Mg when non-basal slip is activated [29].

The interaction between twin and twin is another issue. It is known that more than one variant exist for a certain twin systems. For example, there are six variants for  $\{10\bar{1}2\}$  twin in Mg alloys and three of them are independent. If more than one variant are activated, the twin will intersect with another variant and has the interaction with each other [38-40]. Hong [38] reported that only one variation of extension twin formed when the sample is compressed along the transvers direction of the rolling plane. However, when the sample is under tension along normal direction (throughout the thesis, ND, TD, and RD, refer to normal, transverse, and rolling direction of the plate, respectively), extension twin is activated and more than one variants have been selected in the parent (untwined matrix). The texture and microstructure evolution under these two loading paths are distinctly different due to the difference in the selection of twin variants even though extension twin is the dominant deformation modes in both two cases, which is shown in Figure 2-5. The intersection of twin can inhibit the twin growth and cause stress concentration.

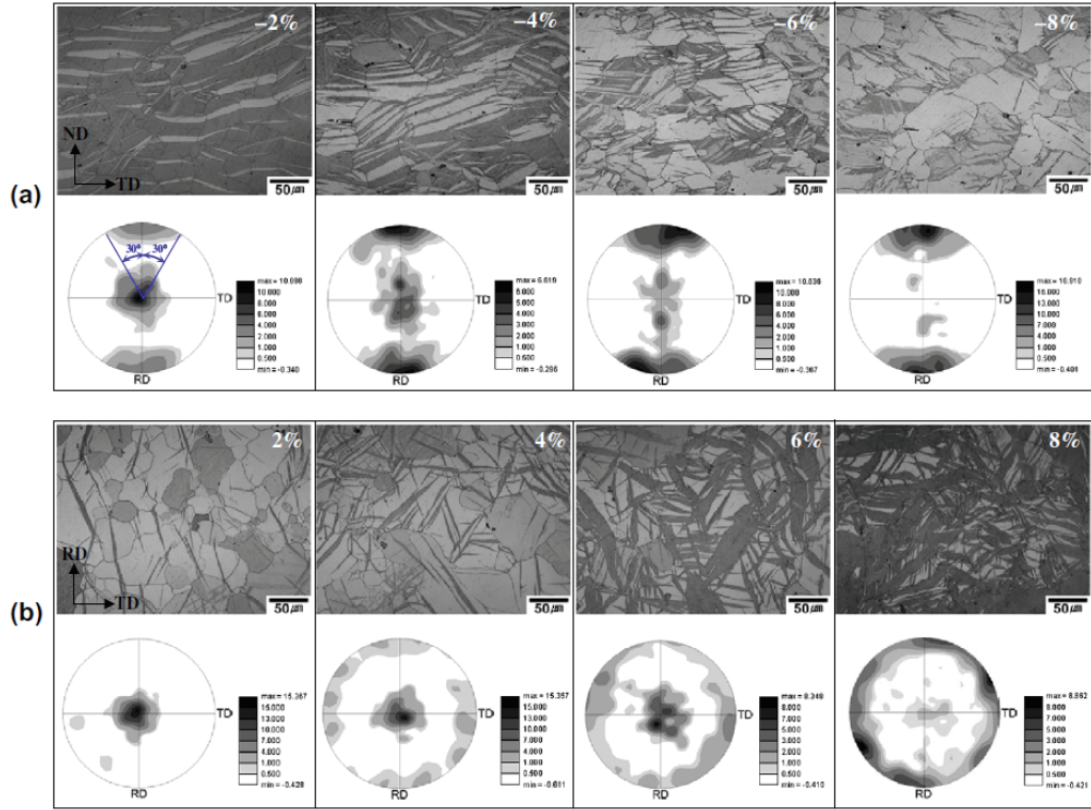


Figure 2-5 Optical microstructures and  $\{0002\}$  pole figures with strain along the: (a) RD, and (b) ND of a hot-rolled Mg plate. The twinned regions are indicated as a bright color in (a) and a dark color in (b). [38]

If the dislocation around the interface can dissolve and form the mobile dislocation in the twin, the stress can be released. On the other hand, the high stress concentration may lead to the crack initiation.

It was found that the twin propagation rate reduces dramatically when two intersecting twins grew equally in a grain, which is the results of the twin-twin interaction [41]. The twin-twin interaction comes from the decrease in twin propagation and growth rate with the increase in the twin nucleation rate. In fact, twin nucleation stress should be higher than the propagation stress according to the general assumptions based on the twinning fundamental mechanisms. The twin propagation is accommodated by the twin dislocation gliding. If twinning disconnections could easily form and glide along the twin boundaries, stress can be relax. Therefore, the nucleation is inhibited by the replacing of the twin propagation and growth. However, if the twin dislocation gliding is substantially impeded by impingements against impenetrable obstacle twins, the local stress increases dramatically due to the stress concentration. Therefore, it can be concluded that twin penetration stress is even higher than the twin nucleation stress, which inhibits the twin propagation and growth when the twin intersects with another twin variant, which is the one effect of the twin-twin interaction. Another type of twin-twin interaction is the Hall-Petch effect. When narrow twin band forms and intersected with the twin with another variant, the dislocation mean-free path reduces.

### 2.2.3.3 The effects of twinning on the hardening behavior

Generally, the strain contributed by twinning is much smaller than that by the slip. However, the twin can provide significantly indirect contribution on the strain as a results of the grain reorientation, release of strain concentration and trigger other types of dislocation, which can show a dramatic influence on the plasticity behavior of Mg alloys [42] and causes the anisotropy in plasticity [43]. Figure 2-6 shows the stress-strain behaviors and the hardening rates for the compression along RD and ND of the hot rolled Mg plate. It was found that the concave-up stress-strain curve shows for the compression along RD case due to the activation of extension twin, while the normal concave-down curve indicates the slip dominant mode in ND compression one. The effect of extension twin on the work hardening of Mg alloys is analyzed [44] based on the diffraction and microstructure analysis. The strain-stress curves of AZ31 extrusion bar under uniaxial compression test at 200°C are shown in Figure 2-7. The extension twin dominates the deformation of Mg alloys and the strain hardening rate as a function of strain (Figure 2-8) indicates that the evolution of extension twin can be separated into four stages. In the stage I, the onset of primary  $\{10\bar{1}2\}$  extension twinning accompanied by macroscopic yielding; stage II, primary twin formation spreads, the slow stress begins to increase, but the work hardening rate remains low; in stage III, the formation of dense secondary  $\{10\bar{1}2\}$  extension twins when different variations of  $\{10\bar{1}2\}$  twin systems are activated, accompanied by an increase in work hardening rate; and stage IV, the saturation of  $\{10\bar{1}2\}$  extension twinning and progressive decrease in the work hardening rate, which finally becomes negative.

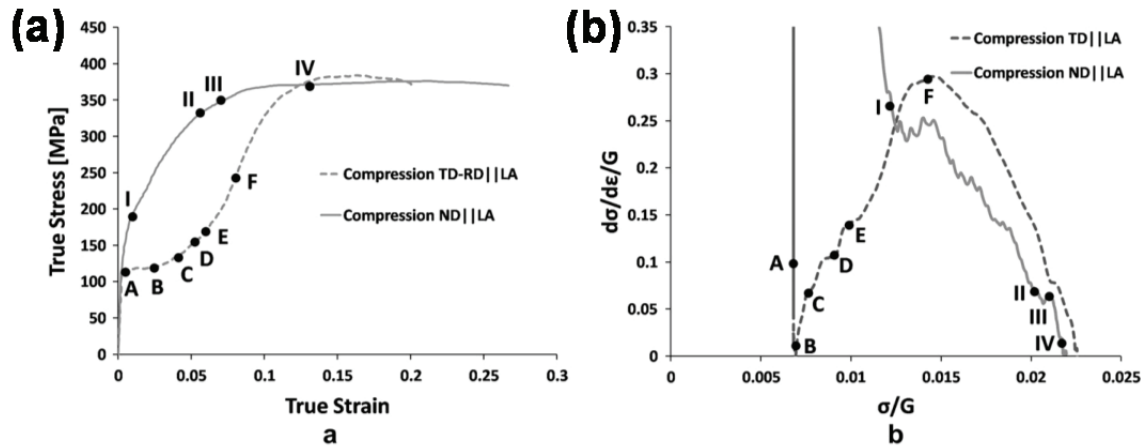


Figure 2-6 Simple compression tests at room temperature on an annealed rolling sheet of AZ31: (a) true stress-true strain responses, (b) normalized strain hardening responses. The various points labeled indicate locations where microstructure investigations were conducted using OM [42].

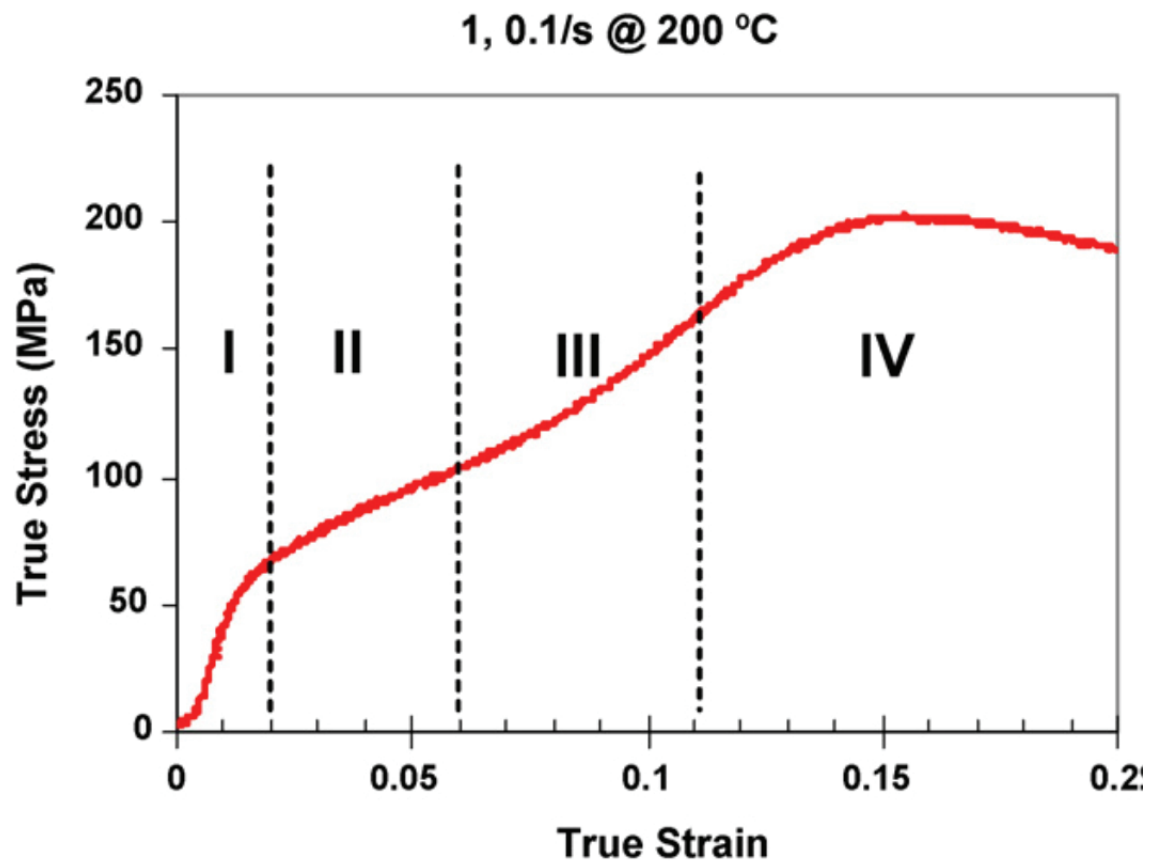


Figure 2-7 The four stages of a representative true strain-true stress curve determined in uniaxial compression of a hot-rolled Mg plate.[44]

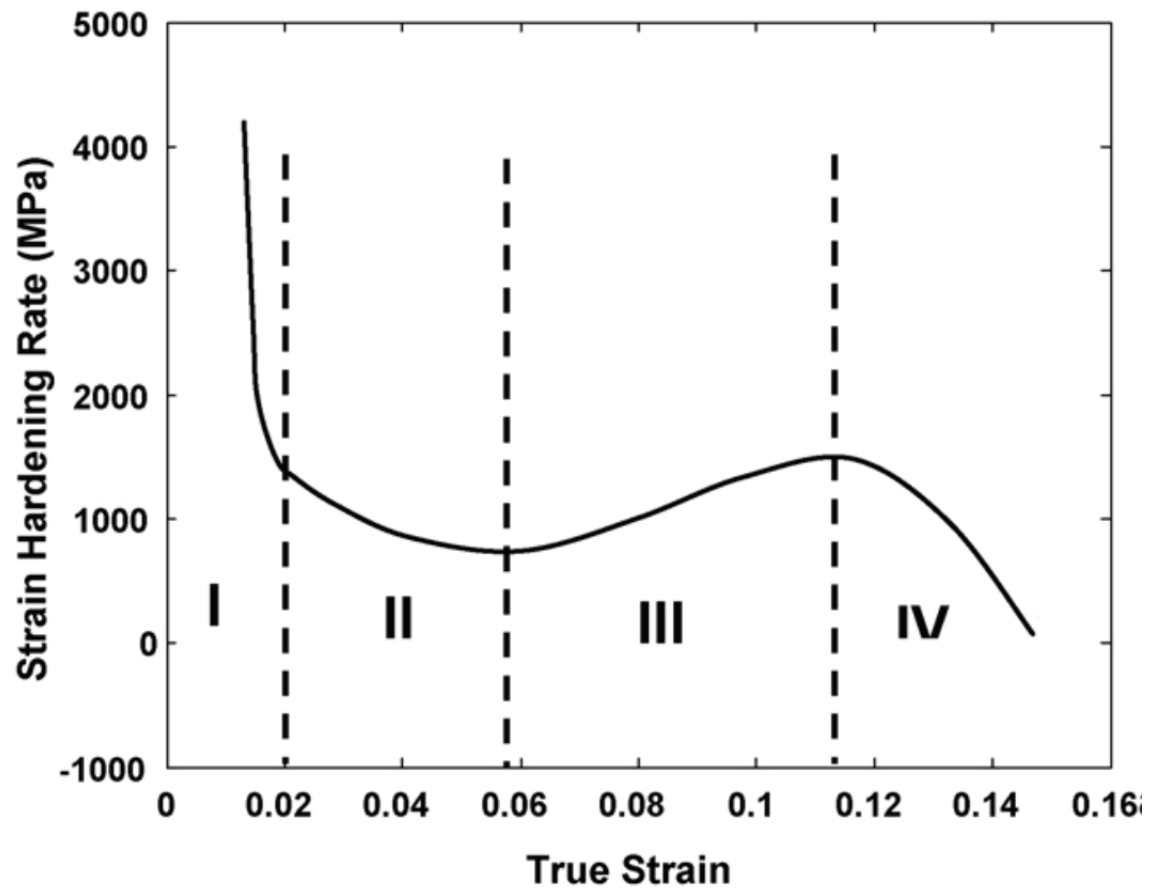


Figure 2-8 The strain hardening rate  $\theta$ - $\epsilon$  curve pertaining to Figure 2-7. [44]

Analysis of twin/matrix misorientation data indicates that the formation of a high density of secondary  $\{10\bar{1}2\}$  twins during state III is responsible for the concave flow curves.

In summary, the twin has three main mechanisms to change the plasticity of Mg alloy [42, 44-51]. The first one is a Hall-Petch-like effect because of the sub-division of twinning. The twinning boundary can be an obstacle for the dislocation movement, which is similar with the grain boundary effects on the dislocation. The second one is the glissile-to-sessile transformation of dislocation already present in the region experiencing the twinning shear transformation (also called Basinski mechanism) [52]. The last and most important one in Mg alloys is crystal reorientation to either a harder or a softer orientation (which is also called texture hardening or softening respectively). The extension twinning can change the crystal orientation by  $86^\circ$  in Mg alloy. For example, the  $\{0002\}$  pole shift from the ND to RD after the deformation under the compression along the RD of the rolled plate, which is shown in Figure 2-9 [53]. However, the qualitatively and quantitatively analysis of the influence of extension twin on the hardening behaviors are still under conflicted.

It was reported by Mahajan [54] that the deformation twin inhibits the movement of dislocation in the FCC crystals, which can explicate the increase in the strain-hardening rate by the decrease in the effective grain size after the twinning occurred. The effect was concurred by Gray [55] in the study of the effect of extension twinning on the hardening in pure Ti. Rohatgi [56] also suggested that the reason why twinning retards the decrease in strain hardening rate is because of the effective grain refinement that results from twinning.



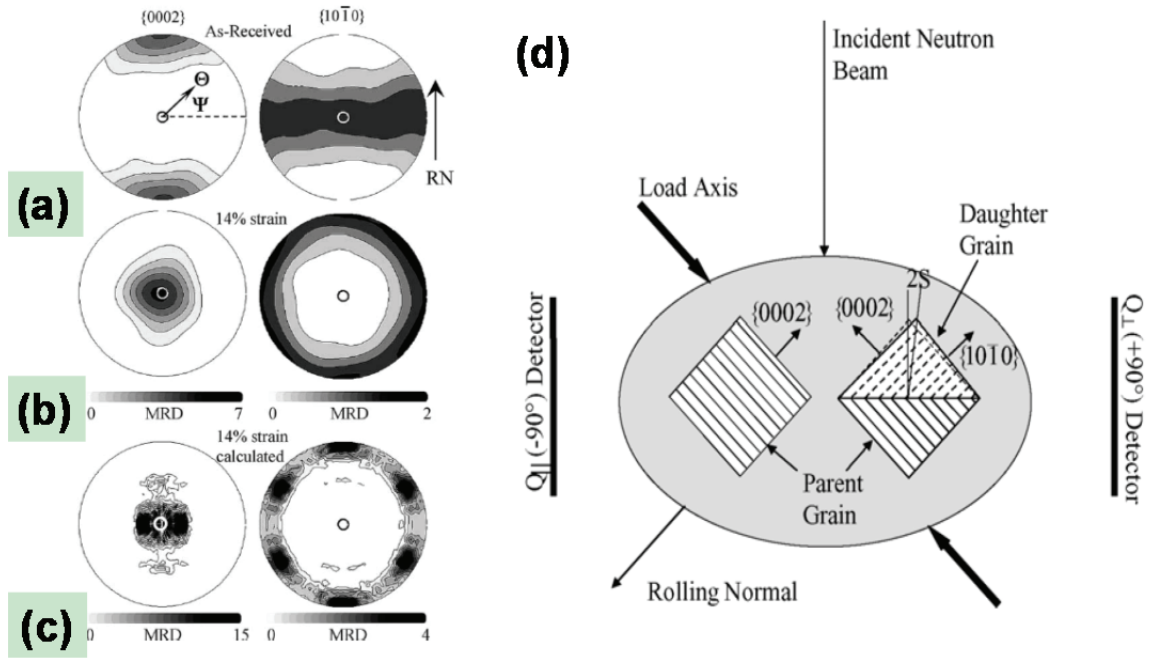


Figure 2-9 Measured texture of rolled magnesium plate: (a) as-received, (b) after 14% in-plane compression, (c) calculated texture after 14% in-plane compression, and (d) Schematic of twinning in magnesium grains in-plane compression sample [53].

Moreover, the Basinski mechanism was verified in the investigation of the extension twinning on the hardening rate in pure Ti as well. Salem [57] reported that the average microhardness numbers in Vickers units in daughter (twinned region) grains were 123 compared with the one in parents as 93, which demonstrated that the daughter is almost 30% harder than the parent. Also, the hardness inside the twins did not show any positive correlation with the thickness of the twin, which indicated that the effect of extension twin in Ti on the hardening rate comes from the Basinski mechanism instead of the Hall-Petch like effect.

On the other hand, Chichili [58] reported that after the low temperature deformation of Ti, the yield strength does not increase obviously compared with the one without the preloading even though the preloading introduced the high twin density in the Ti. This result is in direct conflict with the Hall-Petch and Basinski model, which indicated that twinning should be harder than the parents. Barnett [59] has pointed out that reorientation of the *c*-axis by 90° has a more significant effect on strain hardening than the grain refinement produced by twinning in the *c*-axis extension condition.

The relation between the twin and fracture are complicated. Concerning the association of fracture with twinning, it is stated that twinning and fracture are both essentially cataclysmic processes of strain relief, and the conditions which are ideal for the development of one will generally be satisfactory for the development of the other. This competitive role of twinning against cracking depends closely on the character of the material and defect properties and the experimental conditions [33].

#### 2.2.3.4 Some influence factors for the twin activation

The work of twin can only be positive, which means that the twin can only provide the strain in one direction so that the twin is sensitive to the loading (strain) path. Therefore, the grain orientation has a significant effect on the activation of twin. For instance, the  $\{10\bar{1}2\}$  twin can only be activated when  $c$ -axis is stretched. Moreover, the deformation temperature can also affect the activation of twin. The twin is strain active instead of thermo-active. Therefore, twin is not as sensitive as the slip, especially non-basal slip in Mg alloy to the deformation temperature [35]. When the sample is deformed at low temperature, the non-basal slip systems are not easy to be activated because of the high CRSS. Meanwhile, the pile-up of dislocation along the grain boundaries is more significant because the dislocation climb is very difficult at low temperature. In this case, the twin has high potential to be activated [60]. Considering the relation between the temperature and strain rate, it shows that the twin have high activation with the increase in strain rate. Finally, the grain size can also have the effect on the twin activation. Some reports illustrate that the twin is more likely to happen in the coarse grain instead of the fine grains because the larger dislocation slip length increases the stress concentration at the grain boundaries for the coarse grain [61].

#### ***2.2.4 Critical resolved shear stress (CRSS) of different deformation systems in magnesium and magnesium alloys***

It is known that the minimum shear stress required to initiate slip is called CRSS. When the applied shear stress on the slip plane is higher than CRSS, the single crystal

starts yielding. In magnesium alloy, the CRSSes for slip on different types of slip systems in single-crystal show very wide variations [62, 63]. Slip by  $\langle a \rangle$  dislocations is easiest parallel to the basal planes and prismatic slip. Furthermore, glide of  $\langle c+a \rangle$  dislocations on pyramidal planes is known to be possible in some cases [30]. The relative difficulty of these harder slip modes compared to the softer ones plays an important role in determining textures, strength and ductility [64].

However, for the Mg alloy, single crystal is not easy to be obtained. Therefore, other methods are used to estimate the CRSSes from polycrystal. In polycrystals it is not possible to directly find values of CRSS, although approximate values can be obtained in the case of very strongly textured metals. Estimates can also be made from modeling [65-67], for example, the visco-plastic self-consistent (VPSC) approach, where the results of calculations are compared with experimental data on lattice strain measurements, strength anisotropy or deformation texture evolution.

On the other hand, the CRSSes of different deformation systems are also sensitive to the deformation temperature, which causes the distinct deformation behavior at high temperature [60].

#### 2.2.4.1 CRSS obtained from experiments of single crystal pure Mg

It was reported [68-70] that the basal slip of  $\langle a \rangle$  has very low CRSS at room temperature (about 0.52-0.81MPa) in pure single crystal magnesium and it is stable with the increase in temperature. On the other hand, the CRSS of prismatic slip is much higher than the basal slip, which is up to 40-50MPa. In this case, the ratio of CRSS of prismatic

slip to that of basal slip is close to 48-96. The CRSSes are slightly dependent on the purity of Mg at room temperature deformation. The pyramidal slip is not to be activated at room temperature.

#### 2.2.4.2 CRSS obtained from different modeling methods for AZ31 Mg alloys

However, in Mg alloy, the single crystal is not easy to be obtained for the CRSS measurements. Therefore, some other methods are used to estimate the CRSSes for different deformation modes indirectly. One possible way to estimate the CRSSes is through the fitting the input constitutive parameters in crystal plasticity modeling such as VPSC modeling by comparing with the stress-strain behaviors and texture evolutions obtained through the experiments [22, 71-73]. After optimization of the input constitutive parameters, the absolute or relative values of CRSS can be extracted. When compared with the single-crystal data, the ratios of CRSS values between hard and soft systems are typically seen to be much less (seen examples in Table 2-3) [74]. The simulated CRSS ratios between prismatic and basal slip is in the range of 3 to 8. The deviation in the CRSS ratio is possibly due to the differences in grain size and initial texture distribution. As a result, the mechanical anisotropy of textured hexagonal metals is considerably smaller than that would be expected from simple application of the measured single crystal CRSS values.

Some theories [51] can be used to explain the smaller ratio of CRSS for the polycrystal Mg alloys. In the case of single-crystal Mg, basal dislocation slip dominates the deformation mechanism at room temperature.

Table 2-3 Measurements of single-crystal and polycrystal CRSS values for some important HCP metals obtained from single-crystal experiments and estimated for polycrystalline materials (MPa) [74].

| Materials/method               | Basal <a> | Prism <a> | Pyramidal <c+a> | Ratio hard/soft |
|--------------------------------|-----------|-----------|-----------------|-----------------|
| Mg single crystal              | 0.81      |           |                 |                 |
| Mg single crystal              | 0.76      |           |                 |                 |
| Mg single crystal              | 0.52      |           |                 |                 |
| Mg single crystal              |           | 39        |                 |                 |
| Mg single crystal              |           | 50        |                 |                 |
| Mg single crystal (range)      |           |           |                 | 48-96           |
| Mg-0.1%Zn single crystal       | 1         | 45        |                 | 45              |
| Mg-0.25%Zn single crystal      | 1.2       | 45        |                 | 38              |
| Mg AZ31 polycrystal VPSC model | 45        | 110       |                 | 2.4             |
| Mg AZ31 polycrystal EPSC model | 10        | 55        |                 | 5.5             |
| Mg AZ31 polycrystal EPSC model | 20        | 90        |                 | 4.5             |
| Mg AZ31 polycrystal EPSC model | 30        | 90        |                 | 3.0             |
| Mg AZ31 Sachs Model            | 50        | 100       |                 | 2.0             |
| Mg AM20 small grain EPSC model | 12        | 53        |                 | 4.4             |
| Mg AM30 large grain EPSC model | 2         | 27        |                 | 13.5            |
| Ti-6%Al single crystal         | 190       | 200       | 770             | 4.1             |
| Ti 6/4 polycrystal Sachs model | 444       | 380       | 631             | 1.7             |

However, the situation is different in polycrystalline Mg because of constraint by neighboring grains. If only the basal slip occurs in polycrystals, only two independent slip systems are available out of the necessary five, which causes strain incompatibility at grain boundaries. When the grain boundaries are strong enough, which seems to be the case for most Mg alloys, additional stress arises to maintain strain compatibility at grain boundaries. This compatibility stress gives rise to the activation of nonbasal dislocations as well as twins. This phenomenon has been well documented in bicrystal experiments. Similarly in AZ31Mg alloys, nonbasal dislocations are generated at grain boundaries under compatibility stress. Kobayashi [28] observed dislocations by TEM in equal channel angular extruded (ECAE) AZ31 with an average grain size of 50 $\mu$ m. They found that nonbasal dislocations were generated in the region within several microns from grain boundaries which intersect with basal planes. On the contrary, the generation of nonbasal dislocations was not observed near grain boundaries which are parallel to basal planes. These results indicate that the compatibility effect at grain boundaries restricts the activity of basal slip and, concomitantly, enhances the activity of nonbasal slip. The reduced basal activity and the enhanced prismatic activity lead to the decrease in the CRSS ratio of the prismatic  $\langle a \rangle$  slip to basal  $\langle a \rangle$  slip.

#### 2.2.4.3 CRSS changes as a function of temperature

The CRSSes for different deformation mechanisms decrease with the increase in the deformation temperature [26, 35, 60, 75-78]. The summarized results of CRSSes changed as a function of temperature for different deformation mechanisms are shown in

Figure 2-10. It illustrates that the nonbasal slip are seriously sensitive to the deformation temperature. For example, the CRSS of pyramidal slip decrease ten times from room temperature to 723K. However, the basal slip as well as the extension twin is not very sensitive to the temperature. They almost maintain as a constant or slightly decrease when the temperature increases. On the other hand, the contraction twin seems to be high dependent on the temperature as the nonbasal slip systems. The difference tendencies of the various deformation mechanisms with the changes in temperature lead to the distinct plastic behavior of Mg alloy at high temperature (Figure 2-11). The higher activations of pyramidal slip and contraction twin improve the ductility and decrease the yield and flow stress. The formability of Mg alloys at high temperature is improved by reduction of the r-value, which represent the anisotropic plastic deformation. Therefore, the significant improvement on the processing of Mg alloys at thermal temperature (Figure 2-12).

### **2.3 The influence of texture on the mechanical behaviors**

Texture is another factor to determine the mechanical properties of Mg alloys. It is revealed [79-85] that various textures form after the different forming processes. It is found that the basal plane has the tendency to align to the processing condition in general. For instance, the basal plane is parallel to the rolling plane after the hot-rolling and to the extrusion direction after the hot extrusion. The texture also evolves during the heat treatment because of the recrystallization and recovery. Therefore, the initial texture and texture evolution during the deformation determine the plasticity and mechanical properties of Mg alloys.



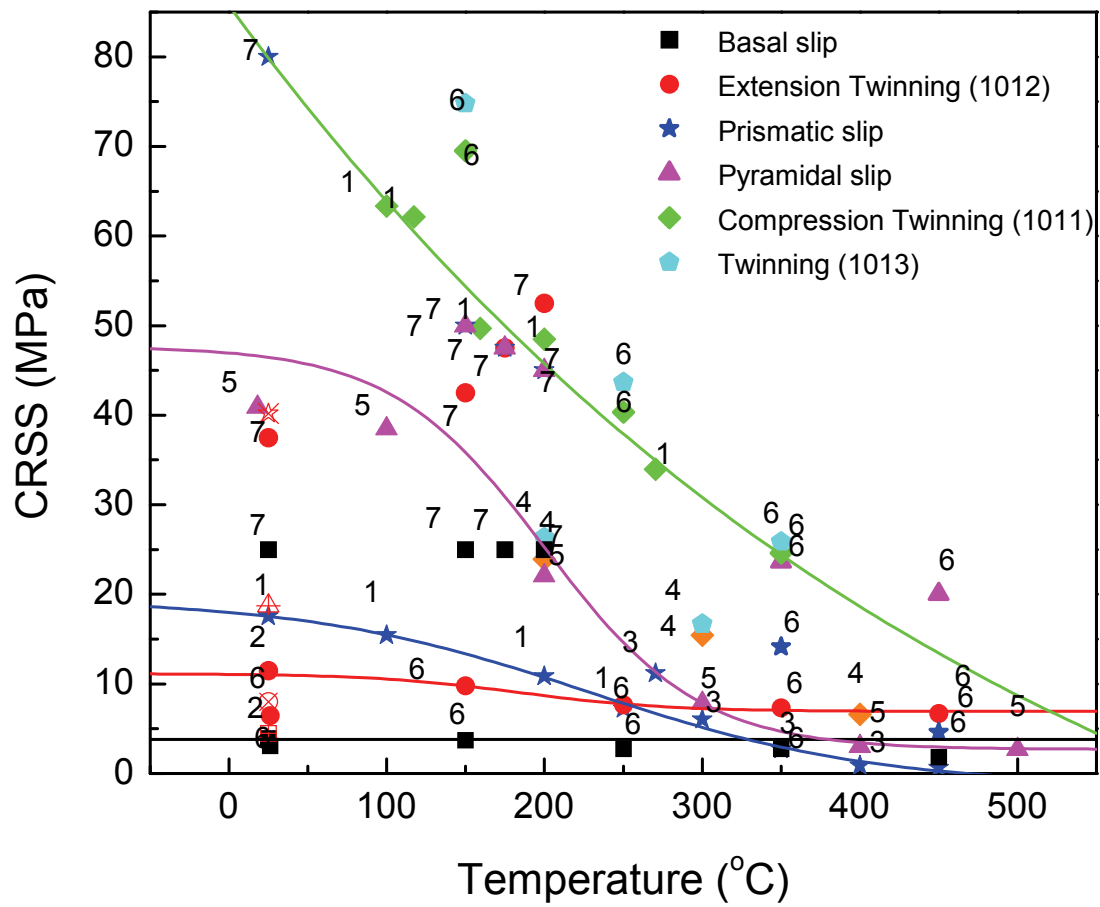


Figure 2-10 CRSSes of different deformation systems as a function of temperature in Mg alloy.

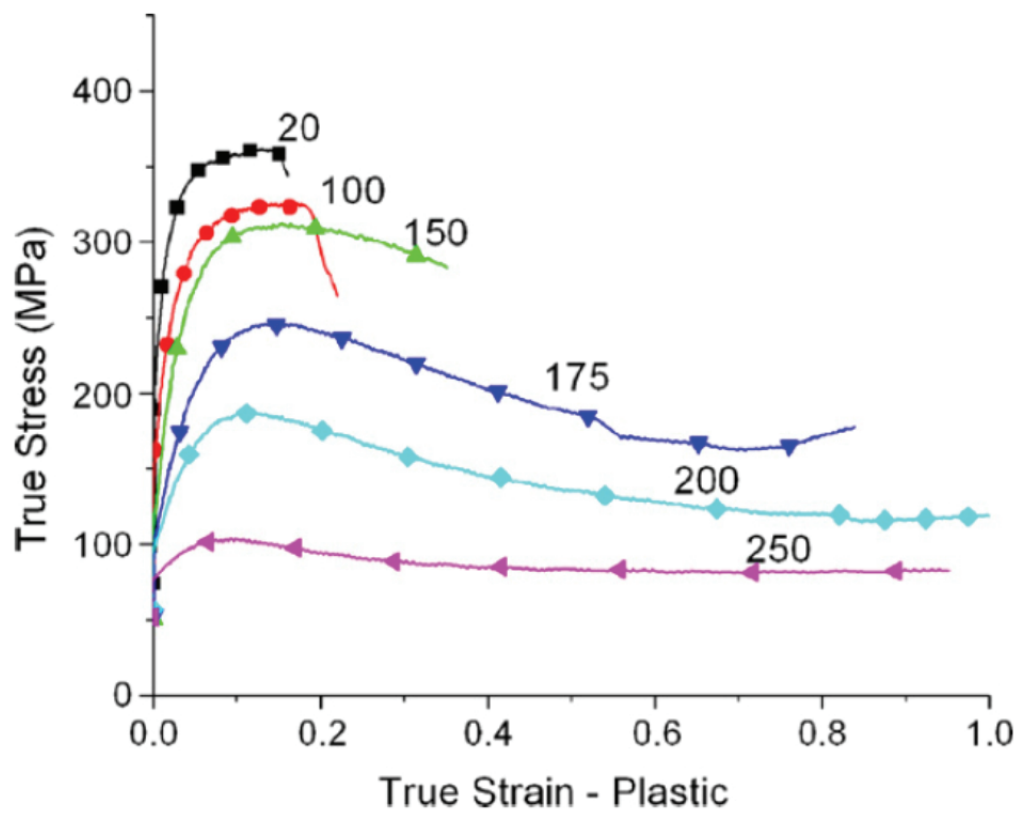


Figure 2-11 Flow curves from ND compression of a hot-rolled Mg plate as a function of temperature [35]

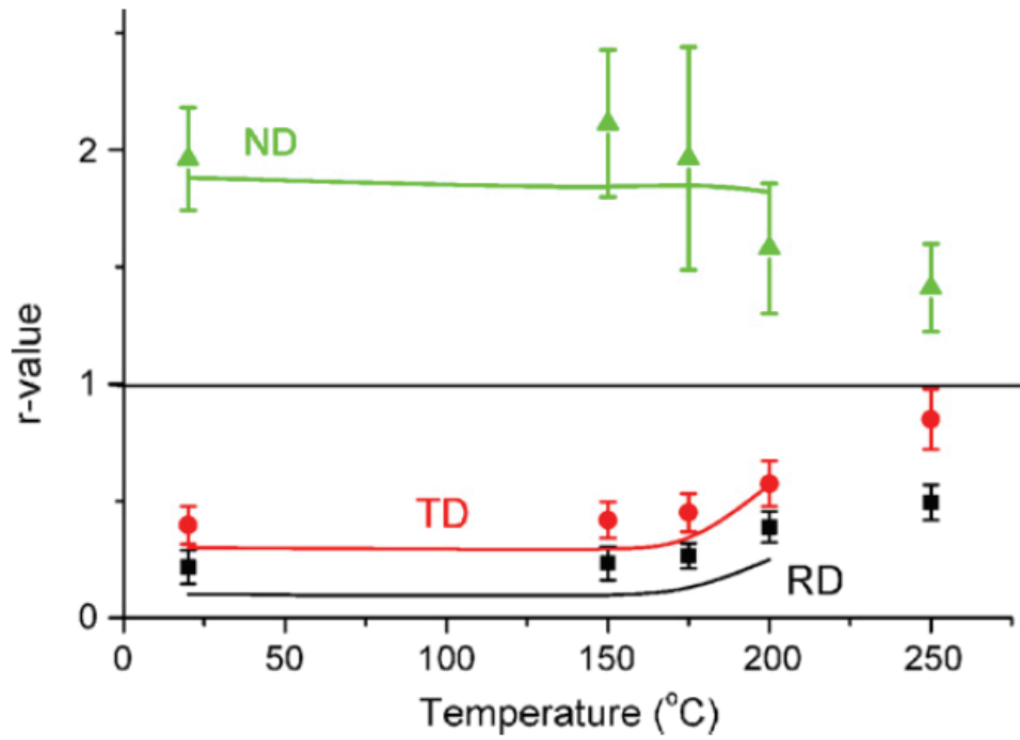


Figure 2-12 Plot of the variation in r-values after compression ( $\epsilon \sim 0.11$ ) at different temperatures of the hot-rolled Mg plate. Symbols represent experimental data and the curves are simulation results [35].

### 2.3.1 Schmid factor in the single crystal of Mg

There is no doubt that the crystal orientation respect to the loading orientation plays an important role for plastic deformation of Mg alloys because of the anisotropy of Mg single crystal. The diversity of possible deformation mechanisms such as basal, prismatic, and pyramidal slip as well as several twinning modes substantially complicates the deformation behavior of hexagonal metals in general. Various authors have provided overviews on the deformation mechanisms in Mg alloys [39, 53, 59, 78, 86, 87].

According to the Schmid law, crystalline slip results from the action of a shear stress on the slip plane. Within the range of stresses in natural situations, the component of stress normal to the slip plane does not influence slip. It can only break the material. Thus the slip process must be considered in terms of the shear stress resolved on the slip plane in the slip direction. Consider a single crystal of cross-sectional area  $A$  under a compression force  $F$  (Figure 2-13). The shear stress resolved in the slip direction is then to be

$$\tau = \frac{F}{A} \cos\lambda \cos\varphi \quad 2-1$$

, where  $\lambda$  is the angle between the slip direction and the force direction, and  $\varphi$  is the angle between the slip plane normal and the force direction. The critical stress required to cause yielding is a function of  $\cos\lambda \cos\varphi$  or the Schmid factor,  $m$ . The slip plane with the greatest resolved shear stress acting upon it will predominate in the slip process. The CRSS for different dislocation slip on distinct slip planes is different. Meanwhile, it is also influenced by the deformation temperature, strain rate, and alloy elements.

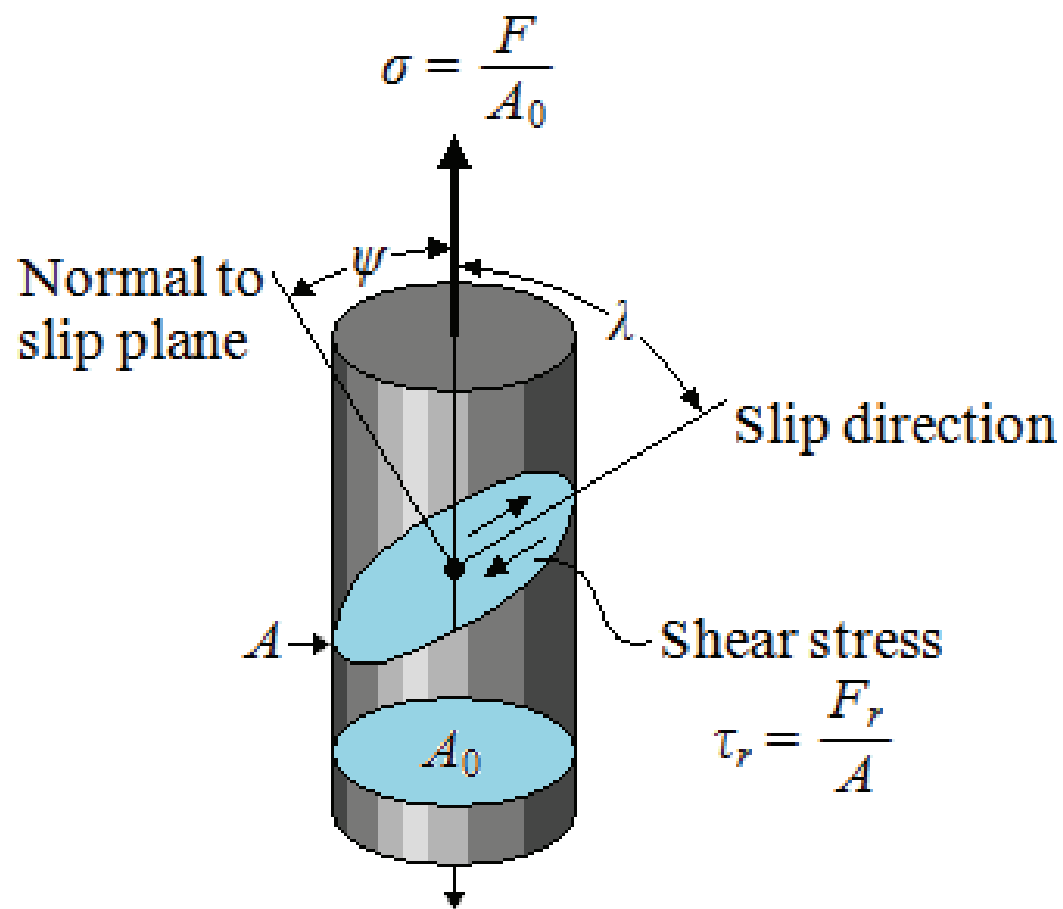


Figure 2-13 Schematics of Schmid law

For the uniaxial tension or compression, the yielding stress,  $\sigma_o$  is equal to minimum value of  $CRSS/m$ , where CRSS is the critical resolved shear stress of different deformation modes and  $m$  is the Schmid factor. The CRSS of different deformation modes for calculation obtained from the literatures [67, 74] used here are shown in Table 2-4. For uniaxial tension or compression, the Schmid factors of possible deformation modes as a function of angle,  $\theta$ , between  $c$ -axis of the crystal and loading direction (illustrated in Figure 2-14) are shown in Figure 2-15. Thus, the  $CRSS/m$  of different deformation modes can be calculated based on the Schmid law, which is shown in Figure 2-16.

Therefore, the extension twinning has the highest activation when tension along  $c$ -axis, while prismatic slip is easiest happen when tension along the basal plane as a combination results of geometry relation and CRSS. On the other hand, under the compression along the  $c$ -axis, the pyramidal and contraction twin have higher potential to be activated, while the normal stress ( $\sigma_o$ ) are much higher than the tension along  $c$ -axis considering the much higher CRSS of contraction twin than the extension twin at room temperature. During the compression along basal plane, the extension twin and prismatic slip can be activated under lower stress. It is clearly demonstrates that the activation of different deformation modes, which have wide range of CRSS values, leads to the anisotropy of plasticity of Mg.

Table 2-4 CRSSs and the ratio of different deformation modes

| <b>Deformation mode</b> | <b>Basal</b> | <b>Prismatic</b> | <b>Pyramidal</b> | <b>Extension Twinning</b> | <b>Compression Twinning</b> |
|-------------------------|--------------|------------------|------------------|---------------------------|-----------------------------|
| <b>CRSS (MPa)</b>       | 10           | 20               | 40.2             | 17                        | 125                         |
| <b>Ratio</b>            | 1            | 2                | 4.02             | 1.7                       | 12.5                        |

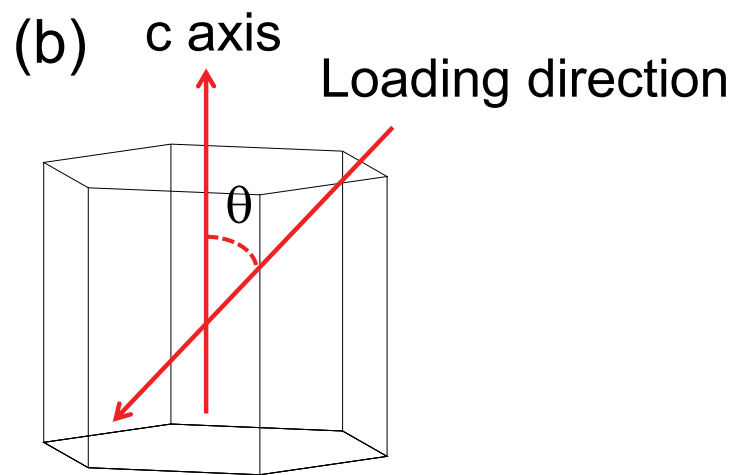
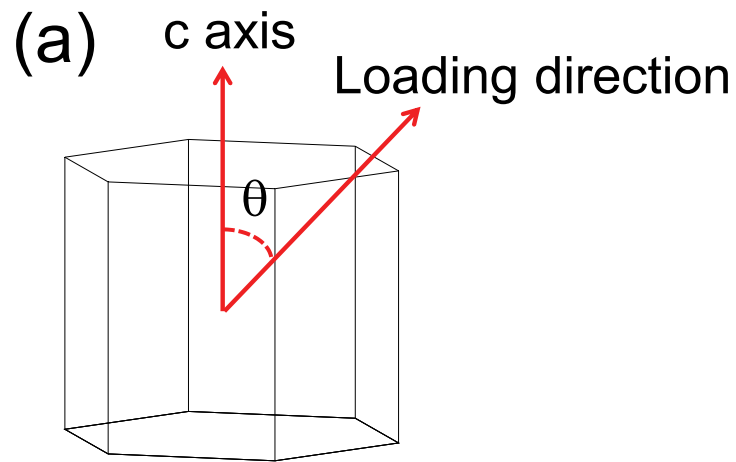


Figure 2-14 The angle,  $\theta$ , between crystal orientation and loading direction for Schmid factor calculation: (a) uniaxial tension, and (b) uniaxial compression



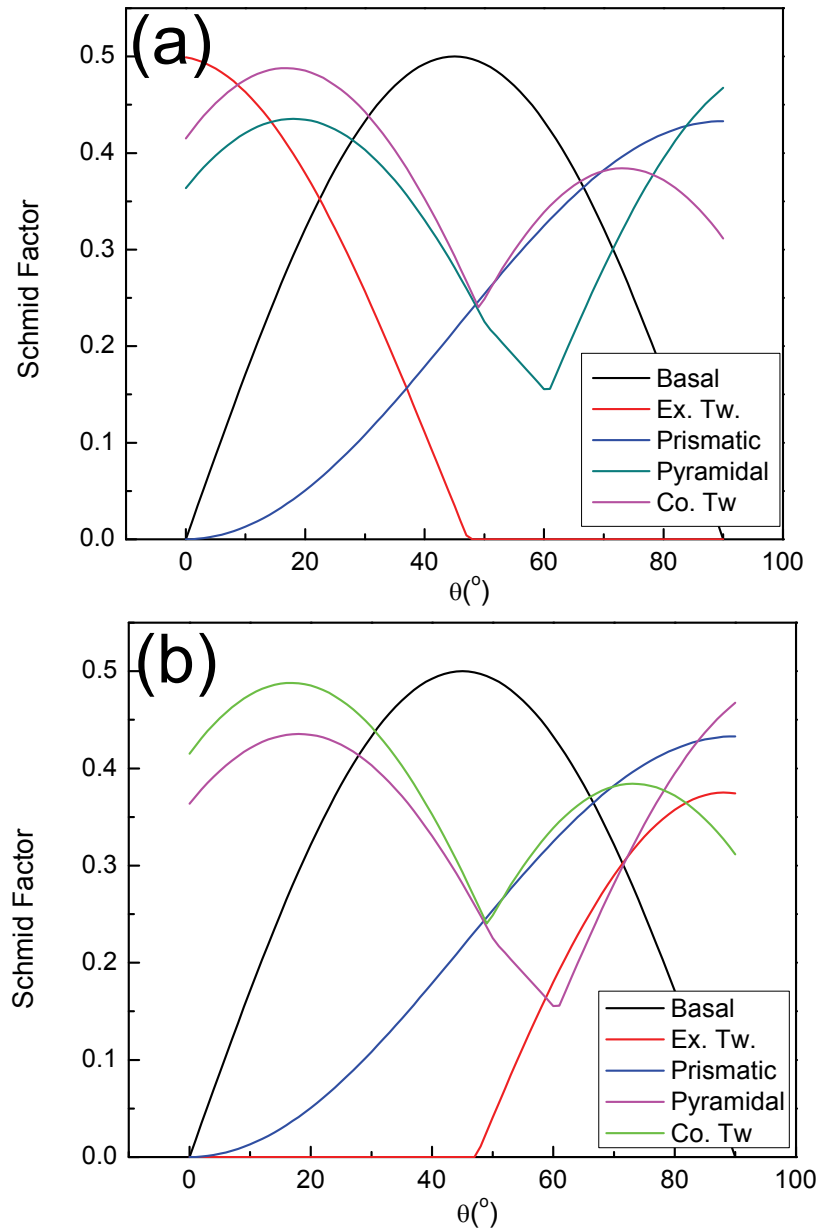


Figure 2-15 Schmid factor,  $m$ , of possible deformation modes at room temperature as a function of angle between crystal orientation and loading direction under different loading paths: (a) uniaxial tension, and (b) uniaxial compression

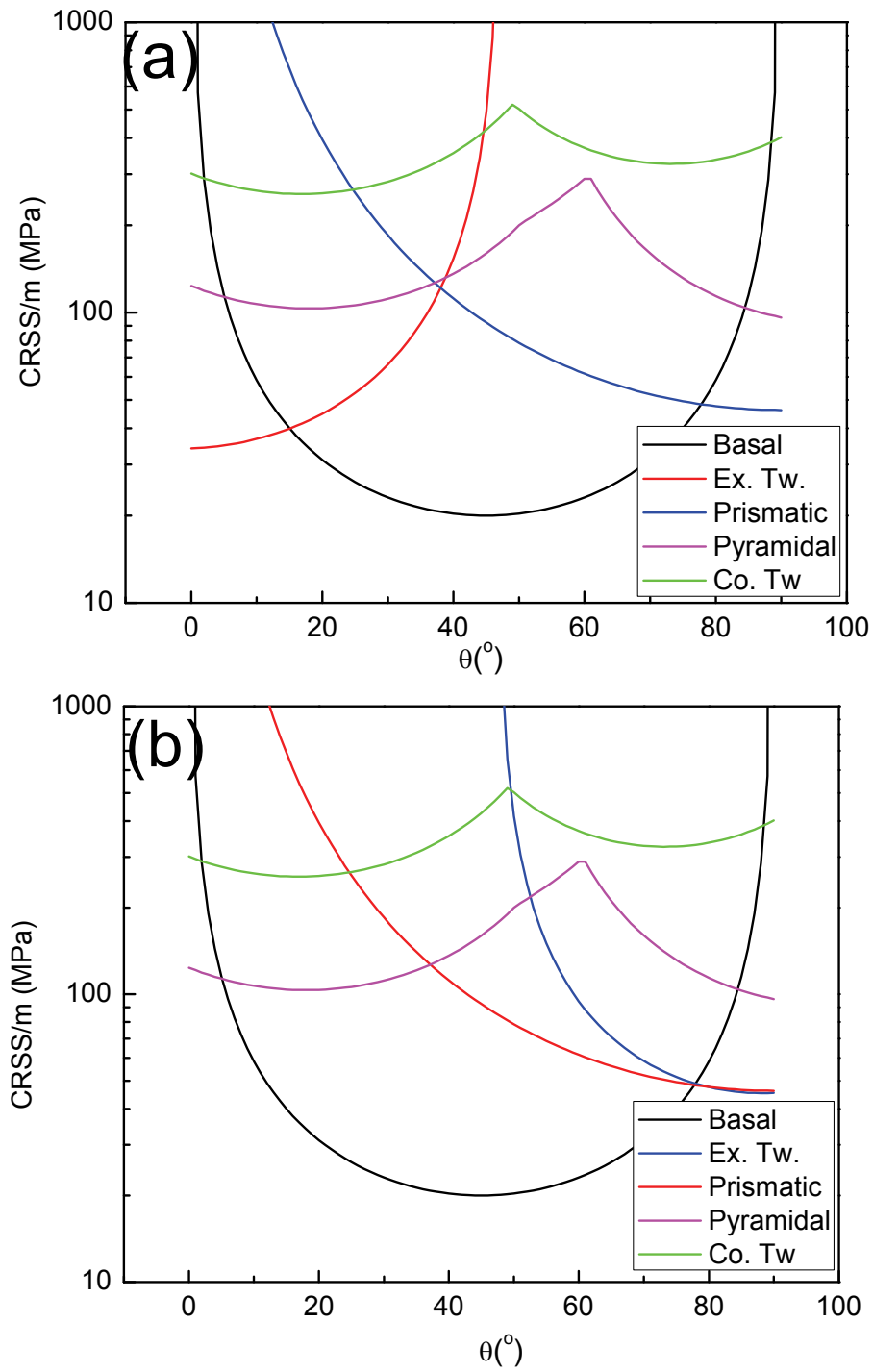


Figure 2-16  $CRSS/m$  of possible deformation modes at room temperature as a function of angle between crystal orientation and loading direction (a) uniaxial tension, and (b) uniaxial compression.

### ***2.3.2 The effect of texture on the activation of various deformation modes in polycrystals***

As a result of the effect of the crystal orientation on the activation of deformation modes, the importance of texture and its influence on plastic deformation was shown for most hexagonal metals, especially for titanium, zirconium and magnesium [24, 88]. Moreover, modeling and simulation of mechanical properties and texture in hexagonal metals taking different slip systems and twinning systems into account has attracted increasing attention [29, 53, 71, 82, 89, 90]. For magnesium and magnesium alloys [22, 42, 43, 46, 48, 86, 91-97] different deformation mechanisms were extensively investigated and all studies consider texture to some extent for discussion of special deformation mechanisms in polycrystals.

Some reports demonstrated that the deformation mechanisms can be obtained with the changes in the initial textures of pure magnesium or Mg alloys and therefore, the materials show distinct plastic deformation behaviors and texture developments. Robert Gehrman [98] chose 4 pure magnesium specimens (Figure 2-17) which have different initial texture (Figure 2-18) by cutting with different directions from a rolling plate. The specimens are compressed through a channel-die device at 100°C, which is illustrated in Figure 2-17b. The flow stresses of these 4 samples are distinct (shown in Figure 2-19) as a result of the different initial textures. The texture measurements and modeling by using RC Taylor mode obviously indicated that different deformation mechanisms are activated with the changes in initial texture.

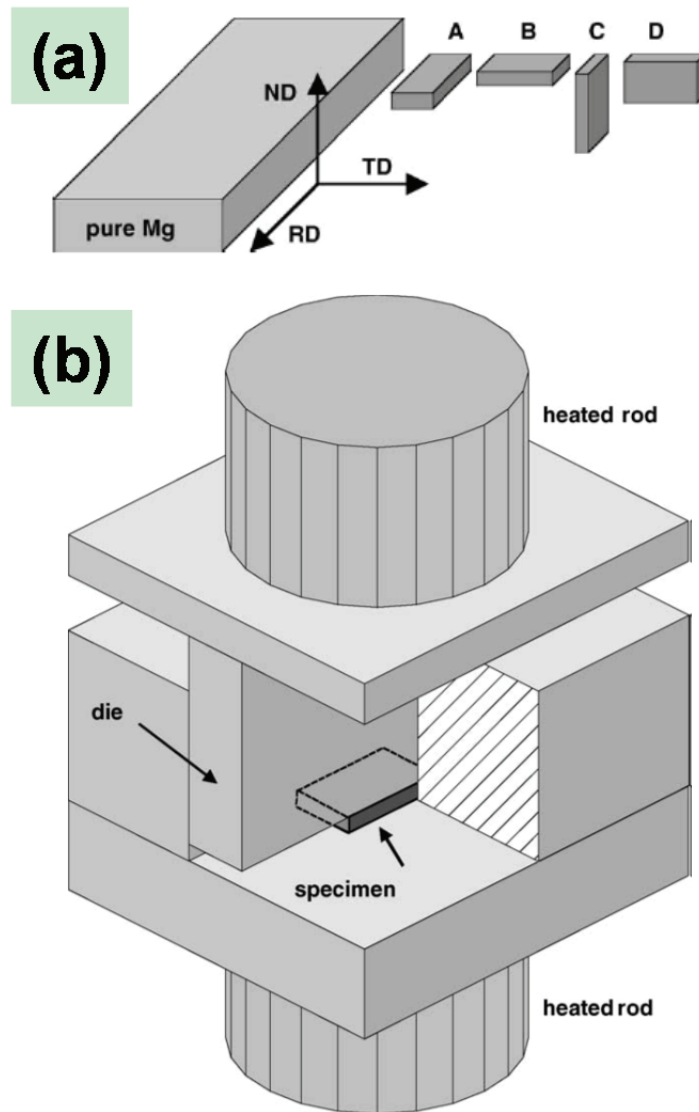


Figure 2-17 Illustration of sample orientation and channel-die compression device (a) Choice of reference system for channel-die experiments; (b) illustration of channel-die compression device. [98]

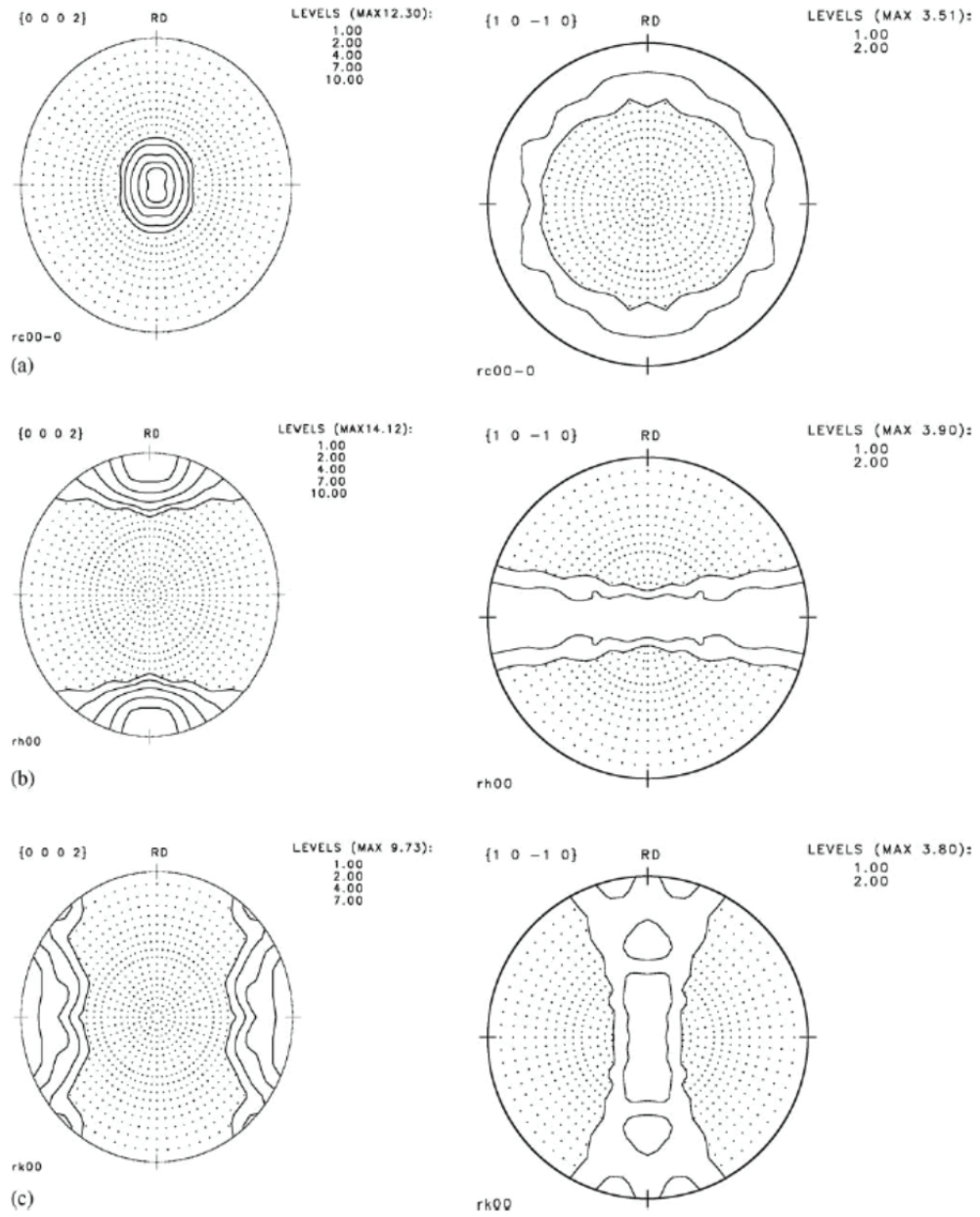


Figure 2-18 Initial textures for channel-die deformation of pure magnesium: (a) case A and B, (b) case C, and (c) case D.[98]

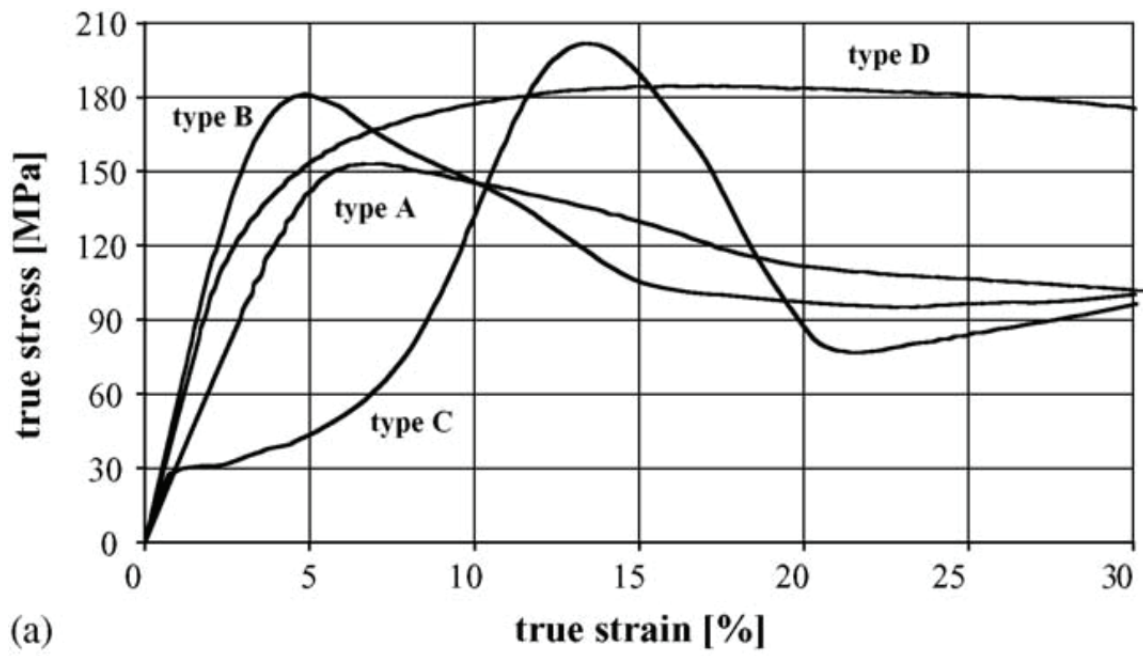


Figure 2-19 Flow curves of different specimen types [98]

In specimen A and B, the basal plane was parallel to the compression plane. During the deformation, only basal slip is activated and forms a  $(30^{\circ}0^{\circ}0^{\circ})$  orientation. When crystallographic slip dominates, rapid initial work hardening is observed and the peak behavior is the more pronounced the less the imposed shape change can be accommodated by crystallographic slip. In case C, the basal slip and extension twin are activated during the deformation. It is indicated that twinning can be initiated at low stresses, and massive twinning leads to little strain hardening because of the low work hardening rate at low strain of case C. After that, the hardening increases dramatically and reaches the peak strain because the basal texture forms after the extension twinning. The sample in case D shows the best ductility when the basal direction was parallel to the compression axis. The prismatic slip is the predominant deformation mechanism in this case and causes very little texture evolution during the deformation.[98]

## **2.4 Grain size effects on mechanical behavior of Mg alloys**

It is well known that the mechanical behaviors of pure metals or alloys are dependent on the grain size followed by H-P relationship. A lot of researches [45, 61, 95, 96] also demonstrated that grain size also has influence on the yield strength of pure Mg or Mg alloys. During the general process of Mg alloys such as hot extrusion, ECAP, FSW, the grain size of Mg alloys changes significantly. Therefore, the analysis of the effects of grain size on the mechanical behavior of Mg alloys can improve the mechanical properties of the wrought Mg alloys.

However, the literature [45, 61, 95, 96, 99-117] shows large discrepancies in the values of H-P parameters ( $\sigma_o$  and  $k_\sigma$ ). It is thought that the texture, solid solution, deformation modes of Mg alloys may have the effects on the H-P parameters. Moreover, not only grain size, but also texture of Mg alloys is change during the process. Thus, the effect of grain size is not straightforward to be measured. It is reported [106] that the inverse H-P relationship during the ECAP of Mg alloys when texture has significant change (Figure 2-20) during the deformation simultaneously.

#### ***2.4.1 Introduction of Hall-Petch relationship***

##### **2.4.1.1 Hall-Petch relation**

Internal grain boundaries present obstacles to the dislocation motion to avoid the passage of a dislocation from one grain to an adjacent one through a grain boundary in the polycrystals. On geometrical considerations, the surface defects such as grain boundaries provide greater resistance to slip than the line or point defects. A model to describe how grain boundaries harden a material is described as follow.

Prior to macroscopic yielding, microscopic yield occurs first in a grain having a favorable slip orientation according to the Schmid law. Therefore, dislocation movement is activated in these grains and pile up against the grain boundary separating it from an adjacent grain which is in the hard orientation. Macroscopic yield cannot occur until the dislocation motion is also activated in such hard adjacent grains.



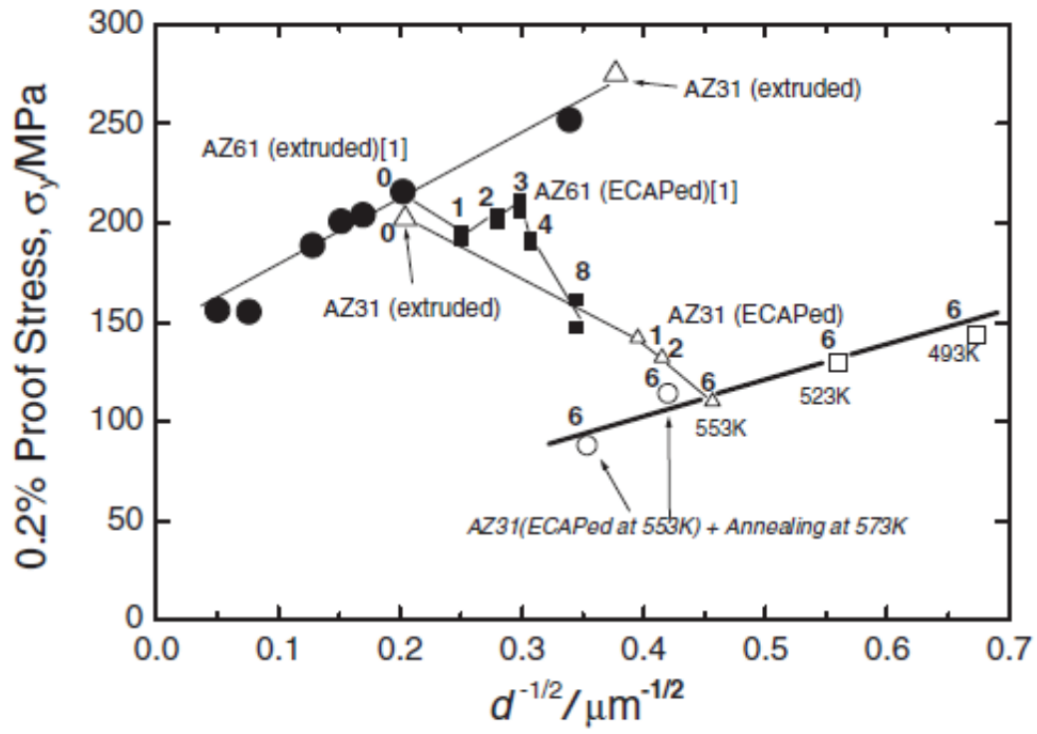


Figure 2-20 The 0.2% proof stress against  $d^{-1/2}$  for the unECAPed and ECAPed AZ61 and AZ31 Mg alloys. Number on the plot denotes the number of pressing [106].

This can be affected by the emission of dislocations from the grain boundaries or, alternatively, the dislocation pileup at the boundary in the deforming grain can produce a stress concentration sufficient to activate slip in the non-deforming grains.

Suppose  $\tau^*$  is the stress required to activate dislocation motion in the unfavorable oriented grain, and these dislocations are located a distance  $r$  from the boundary. The stress concentration due to the dislocation pileup in the active grain increases with the number of dislocations that come up against the impenetrable boundary. In turn, this number increases as the mean slip distance (here is the grain size,  $d$ ) in the active grain increases. Analysis shows that dislocation activation in the second grain occurs when

$$(\tau_{app} - \tau_o) \left( \frac{d}{4r} \right)^{1/2} = \tau^* \quad 2-2$$

Here,  $\tau_{app}$  is the applied shear stress at which this activation occurs and  $\tau_o$  is the intrinsic stress resisting dislocation motion in the deforming grain. The parameter  $(d/4r)^{1/2}$  represents the stress concentration arising from the pileup and it increases with the number of dislocations in it. Rearrangement of Equation 2-2 allows the applied shear stress to be expressed in terms of the grain diameter

$$\tau_{app} = \tau_o + \tau^* r^{1/2} d^{-1/2} = \tau_o + k'_y d^{-1/2} \quad 2-3$$

In terms of yield strength, it can be rewritten as

$$\sigma_y = \sigma_o + k_y d^{-1/2} \quad 2-4$$

This is known as the H-P equation and it indicates that the yields strength of a polycrystal increases linearly with  $d^{-1/2}$  [105].

The degree to which grain-size hardening can be utilized depends on the material's H-P coefficient. The H-P parameters of some common alloys are shown in Table 2-5 [105]. Generally, the FCC structure has a relatively lower H-P coefficient comparing with the HCP and BCC structures.

#### 2.4.1.2 The friction stress, $\sigma_o$

In the H-P relation (Equation 2-4),  $\sigma_o$  is the friction stress for dislocation movement, which is similar to the yield strength of the single crystal. From the relation of  $\sigma = m\tau$  ( $\sigma$  as normal stress and  $\tau$  as shear stress),  $\sigma_o$  is given by

$$\sigma_o = M\tau_{CRSS} \quad 2-5$$

where  $M$  is the Taylor orientation factor and  $\tau_{CRSS}$  is the single crystal CRSS [99].

For a single crystal, the yield stress  $\sigma_{0.2}$  is given by  $\sigma_{0.2} = (1/m)\tau_{CRSS}$ , where  $m$  is Schmid factor. For the cast Mg alloys, which have a random crystal orientation (shown in Figure 2-21), the Schmid factor  $m$  for basal slip can be calculated as [109]:

$$m = \left(\frac{1}{\pi^2}\right) \cdot \left(\frac{2}{\pi} \int_0^{\pi/2} \frac{3}{\pi} \cos\chi \sin\chi dx\right) \approx 0.031 \quad 2-6$$

However, it worth to note that the different deformation modes may be activated when the initial texture of Mg alloys are various. Considering the wide discrete of the CRSS for distinct deformation modes,  $\sigma_o$  is highly dependent on the deformation modes and texture of Mg alloys.

Table 2-5 values of  $k_y$  for some common alloys[105]

| <b>Material</b>  | <b>Crystal Structure</b> | <b><math>k_y</math> (<math>MPa \cdot mm^{-1/2}</math>)</b> |
|------------------|--------------------------|------------------------------------------------------------|
| Low-carbon steel | BCC                      | 9.7                                                        |
| Armco iron       | BCC                      | 18.4                                                       |
| Molybdenum       | BCC                      | 55.9                                                       |
| Zinc             | HCP                      | 7.0                                                        |
| Magnesium        | HCP                      | 8.8                                                        |
| Titanium         | HCP                      | 12.7                                                       |
| Copper           | FCC                      | 3.5                                                        |
| Aluminum         | FCC                      | 2.2                                                        |

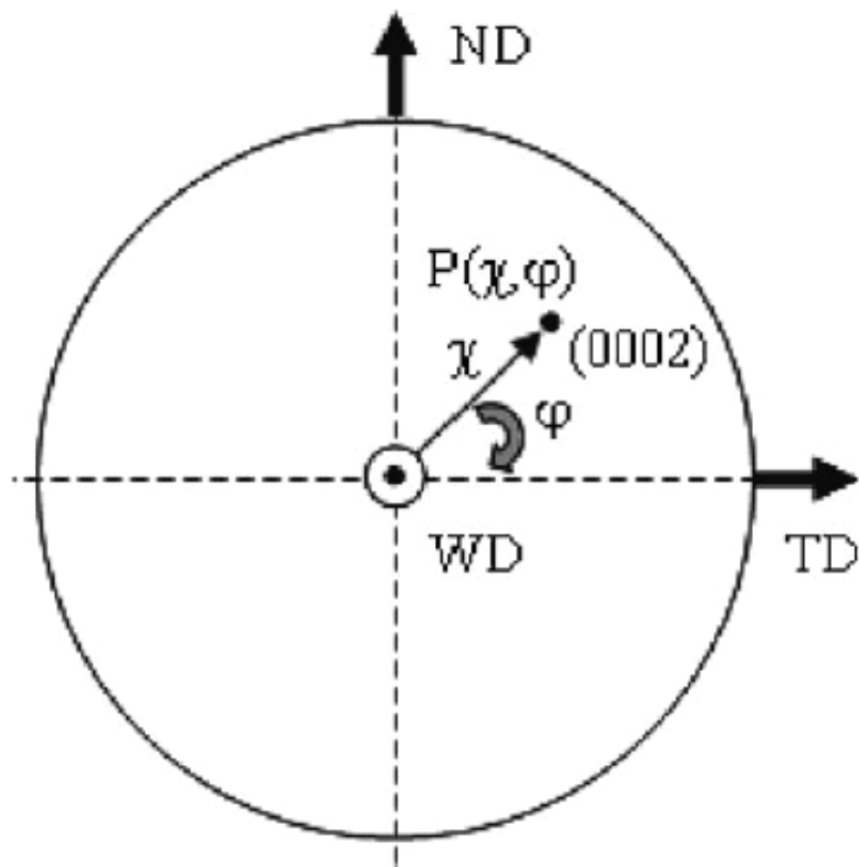


Figure 2-21 Schematic drawing showing the position of (0001) slip plane of a grain with respect to the stress axis in the stereographic projection [109].

#### 2.4.1.3 The strength coefficient, $k_y$

According to the Equation 2-4,  $k_y$  is the characteristic constant representing the resistance to dislocation motion provided by the grain boundaries. The definition of  $k_y$  in Equation 2-4 is as follows according to Armstrong [118].

$$k_y = m^2 \tau_{CRSS} r^{1/2} \quad 2-7$$

, where  $\tau_{CRSS}$  is the critically resolved shear stress for slip,  $m$  is the orientation factor related to the activated slip system and  $r$  is the distance from the nearest dislocation pileup to the dislocation source in the adjacent grain.

Therefore, it is clear to show that the H-P parameters are related to the activated deformation mode and crystal orientation based on the Equation 2-5 and 2-7, which change with the initial texture of alloys.

### 2.4.2 *The variation of H-P parameters of AZ31B Mg alloys*

#### 2.4.2.1 The H-P parameters from literature data of AZ31B with different proccession

Even though the concentration of alloys does not change in the same alloy, the H-P parameters of Mg alloys are various considering the high dependence of the initial texture and deformation modes. Wide discrepancies in the values of H-P parameters ( $\sigma_o$  and  $k_y$ ) are summarized in Table 2-6 [45, 61, 95, 96, 99-117].

Table 2-6 indicates that the grain size has the effects on the yield strength of Mg and Mg alloys, which follows H-P relationship in a wide range of grain size (from 2 $\mu$ m to 1500 $\mu$ m).

Table 2-6 Hall-Petch parameters ( $\sigma_o$  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.

| Mat. | Processing Condition  | Loading Path       | Grain Size ( $\mu\text{m}$ ) | $\sigma_o$ (MPa) | $k_\sigma$ (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.

Moreover, when the grain size decreases, the yield strength of Mg increases as a results of the positive sign of strength coefficient,  $k_y$ , as an example shown in Figure 2-22. However, the H-P parameters including  $\sigma_o$  and  $k_y$  are various when materials processing or test condition are different. For instance, the processing methods such as rolling, ECAP, and FSP changes the H-P parameters. 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 [119]. The H-P parameters are particularly sensitive to the loading path, which is illustrated by comparing tension [108] and compression [113] along the extrusion direction (ED). The strength coefficient  $k_y$  ( $\text{MPa} \cdot \text{mm}^{1/2}$ ) varies from 5.0 to 12, while the friction stress  $\sigma_o$  (MPa) ranges from 10 to 131. Therefore, it is clear that in some condition, grain size has relatively high effect on strength hardening.

#### 2.4.2.2 The weak grain size sensitivities of FSWed and ECAPed AZ31B alloys

Some reports demonstrate that the grain size effects on the strength of Mg alloys are weak, which is also shown in Table 2-6. For instance, compared with the samples processed by rolling and extrusion, the FSWed samples shows much lower strength coefficient ( $k_y = 5.1 \text{ MPa} \cdot \text{mm}^{-1/2}$ ), which is shown in Figure 2-23 [108]. It is indicated that the main reason for the difference of the H-P parameters is the change in texture. Figure 2-24 illustrates the obviously difference of the initial texture for the hot extrusion sample and FSPed sample.



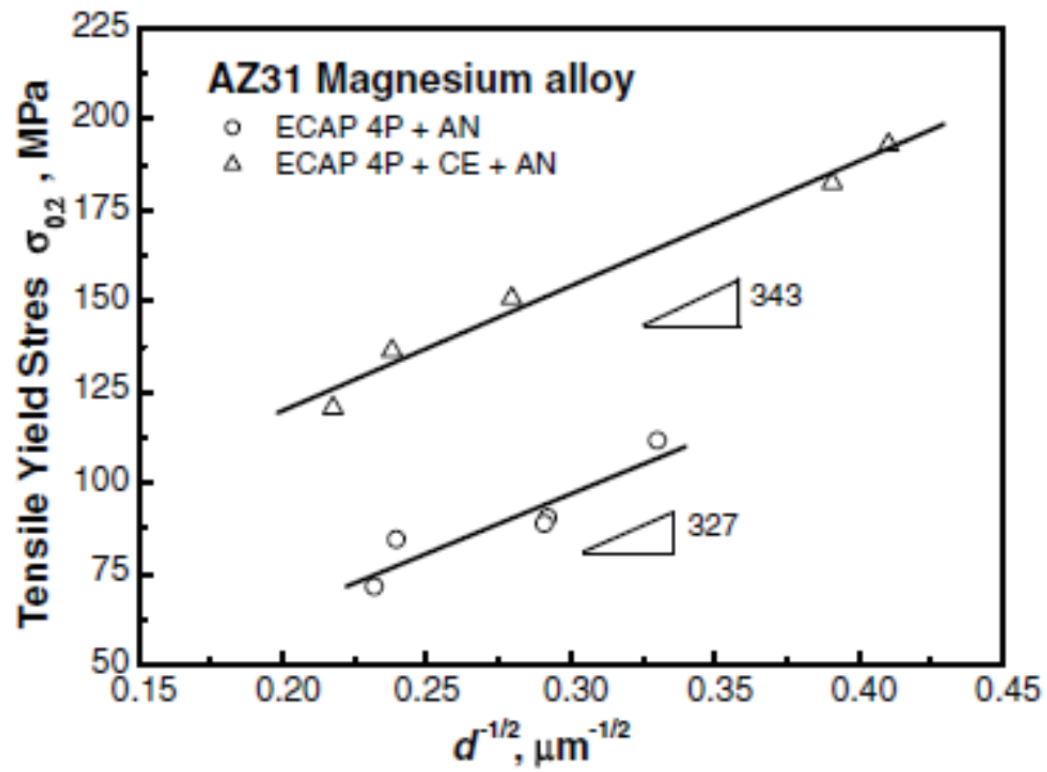


Figure 2-22 H-P relation from two groups of samples of AZ31alloy with substantial difference in texture [112].

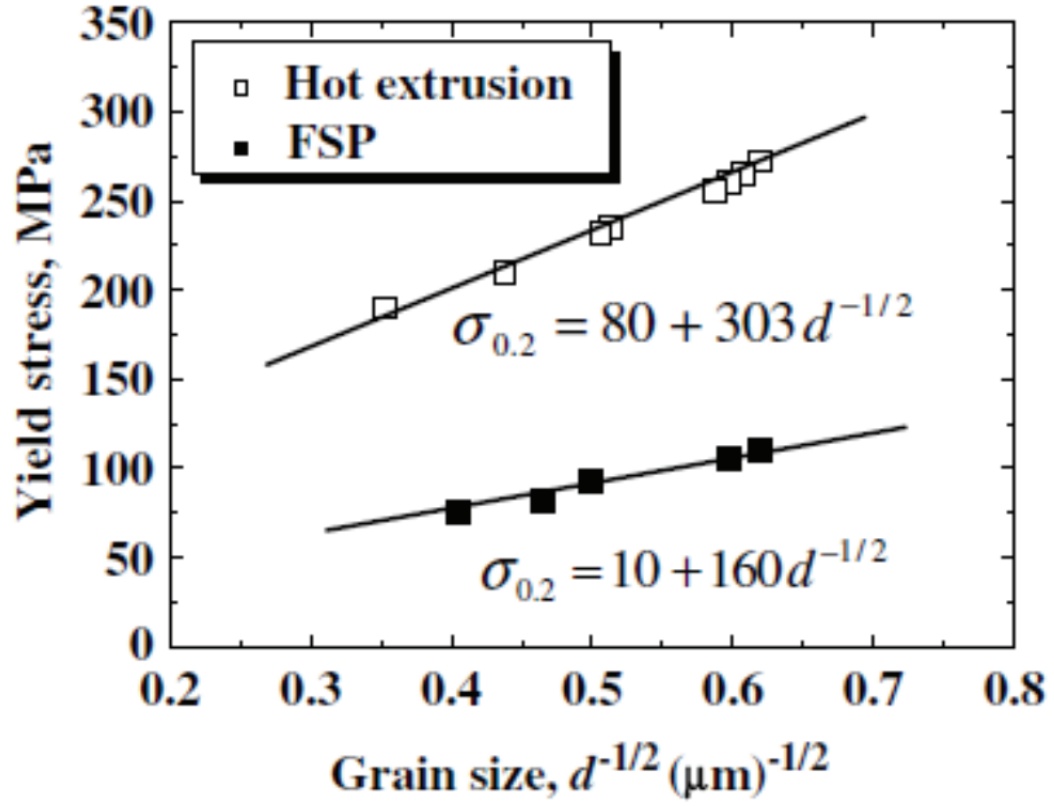


Figure 2-23 H-P relationship for the extruded and FSPed specimens at room temperature [108]

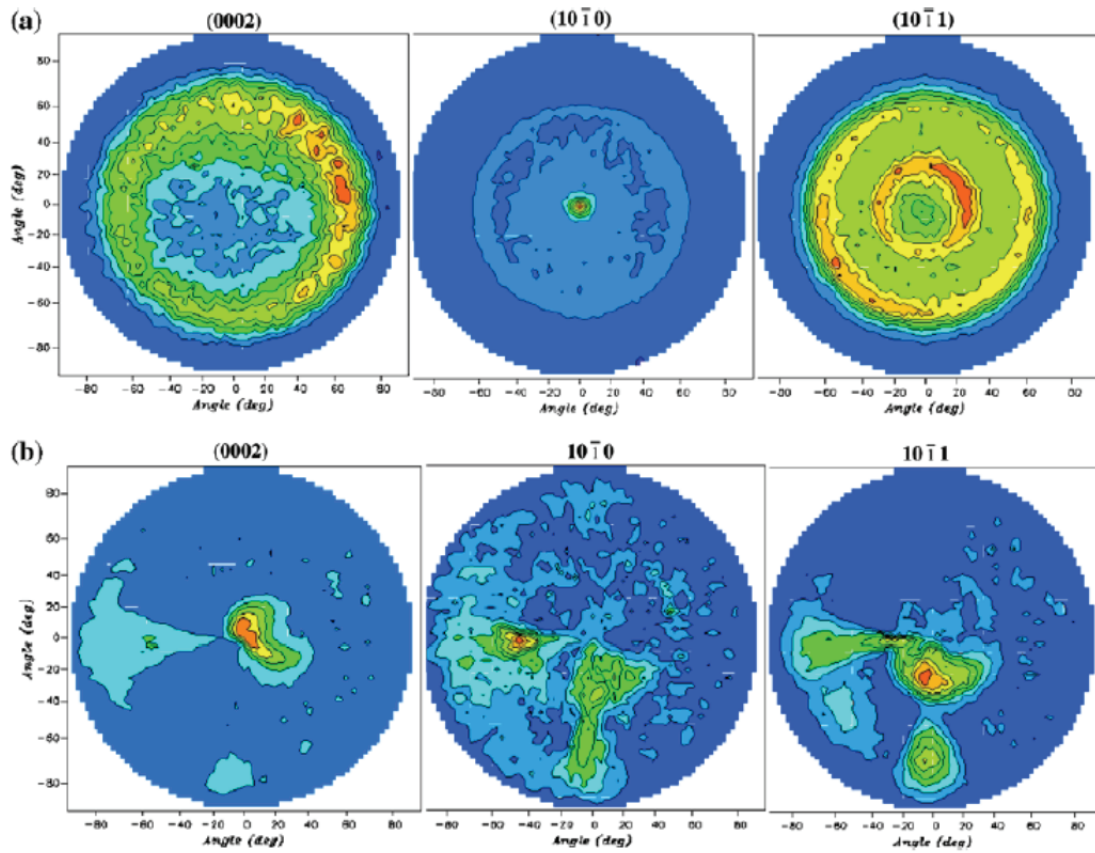


Figure 2-24 X-ray pole figures show the texture in: (a) hot extruded, and (b) FSPed AZ31 samples seen from the transverse cross-sectional plane, namely, the working (extrusion or welding) direction of the specimen is parallel to the normal of the pole figures. For the FSP pole figures the ND and TD are vertical and horizontal, respectively.[108]

After the FSP, the  $\{0002\}$  rotates from the direction perpendicular to the ED to the direction parallel to the work direction. The tensile direction is parallel to the extrusion or FSW advancing direction, respectively. Thus, the Schmid factor,  $m$ , for  $\{0002\} < 11\bar{2}0 >$  basal slip system, which has the lowest CRSS at room temperature, for the hot extrusion sample are close to 0. However, in the FSWed sample, the  $m$  can be calculated as follows according to the schematic drawing of basal planes distribution in Figure 2-25:

$$m = \left(\frac{1}{\pi}\right) \cdot \left(\frac{3}{\pi} \int_0^{\pi/2} \frac{3}{\pi} \cos\chi \sin\chi dx\right) \approx 0.3 \quad 2-8$$

Therefore, basal slip is much easier to be activated in the FSWed sample than in the extrusion one, which may cause the lower value of  $\sigma_o$ . Meanwhile, as mentioned above, the grain size dependence,  $k_y$ , expresses the magnitude of the resistance of the grain boundaries as an obstacle to the slip across the grain boundaries, which is related to the stress concentration originating from the dislocation pile-ups behind the grain boundaries. Furthermore, the stress concentration against the grain boundaries affects the dislocation action of the neighboring grain. When the neighboring grain has a preferred orientation (referred to here as a soft orientation with a high orientation factor) with regard to the applied stress, the action of the dislocations in the soft oriented grain becomes easier than that in the non-preferred oriented grain (referred to here as a hard orientation with a low orientation factor) with regard to the applied stress. Thus, in soft oriented grains, the stress multiplication in the next grain should be much lower than that in the hard oriented grains.

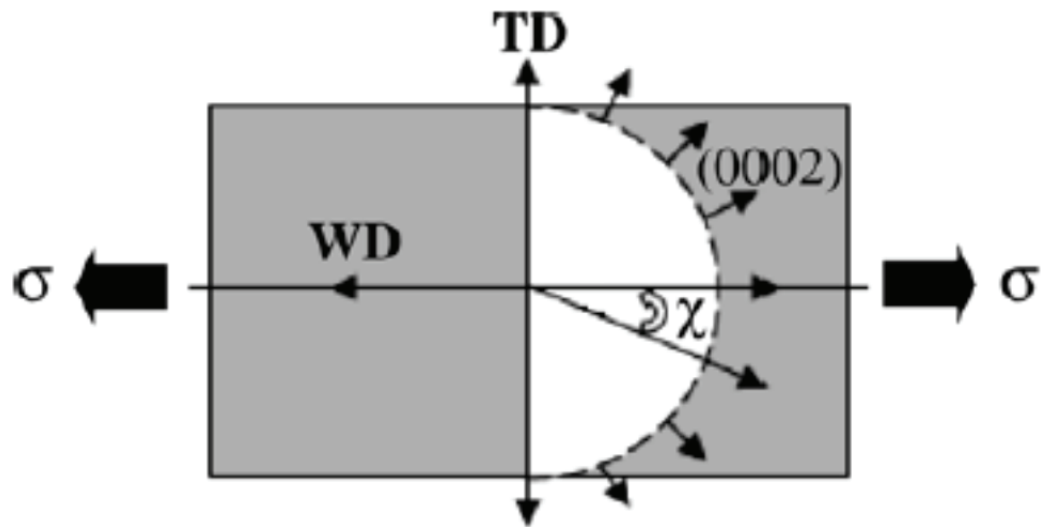


Figure 2-25 Schematic drawing showing the  $\{0002\}$  slip planes distribution in the stir zone. Scattering arrows indicate the ND of the  $\{0002\}$  basal planes [108].

This means that, under the condition of the same grain size, the resistance of the grain boundaries as an obstacle to slip across the grain boundaries in the FSP samples (soft orientation) is much lower than that in the extruded samples (hard orientation), which results in the low  $k_y$ . Thus, both  $\sigma_o$  and  $k_y$  would be low and in turn result in the low yield stress in the FSP samples as compared to the extruded specimens.

The similar results also obtained from the ECAPed Mg alloys. The H-P relation of Mg AZ31B alloy with different ECAPed pass is shown in Figure 2-20 [106]. Comparison of yield strength of un-ECAPed extrusion bar, 1 pass, 2 pass and 6 pass ECAPed samples, yield strength increase with the grain coarsen, which means the “abnormal” inverse H-P relation is obtained. However, comparing the samples all processed by 6 pass ECAP, the positive H-P slope is shown with the strength coefficient as  $5.7 \text{ MPa} \cdot \text{mm}^{-1/2}$ .

The extrusion bar which is the un-ECAPed sample has the strong basal fiber texture. The  $\{0002\}$  planes are almost parallel to the extrusion direction. For the tension along the extrusion direction, the basal plane is very hard to be activated. On the other hand, the texture has a significant change during the ECAP. The texture of the sample with 6 pass ECAP at different temperature are shown in Figure 2-26. The Schmid factor of basal slip and prismatic slip are calculated based on the texture measurement, which is shown in Table 2-7 [106].

Thus, it can be concluded that during the ECAP, the texture changes gradually to increase the Schmid factor of basal slip.

Table 2-7 Textures and Schmid factor values for the ECAPed AZ31 alloys.[106]

| ECAP pass no. | ECAP temp.                        | Texture                          | Schmid factor   |                 |
|---------------|-----------------------------------|----------------------------------|-----------------|-----------------|
|               |                                   |                                  | basal           | prismatic       |
| unECAPed (0)  |                                   | ED//[10 $\bar{1}$ 0]             | 0; 0; 0         | 0; 0.43; 0.43   |
| 6             | 553K                              | (01 $\bar{1}$ 2)[ $\bar{2}$ 243] | 0; 0.432; 0.432 | 0; 0.203; 0.203 |
| 6             | 523K*                             | (02 $\bar{2}$ 3)[ $\bar{4}$ 154] | 0; 0.423; 0.423 | 0; 0.264; 0.264 |
| 6             | 493K                              | (02 $\bar{2}$ 3)[ $\bar{4}$ 154] | 0; 0.423; 0.423 | 0; 0.264; 0.264 |
| 6             | 523K*+annealing at 573K for 20min | (01 $\bar{1}$ 2)[ $\bar{2}$ 243] | 0; 0.432; 0.432 | 0; 0.203; 0.203 |
| 6             | 523K*+annealing at 573K for 80min | (01 $\bar{1}$ 2)[ $\bar{2}$ 243] | 0; 0.432; 0.432 | 0; 0.203; 0.203 |

\*ECAPed to 2 passes at 553K

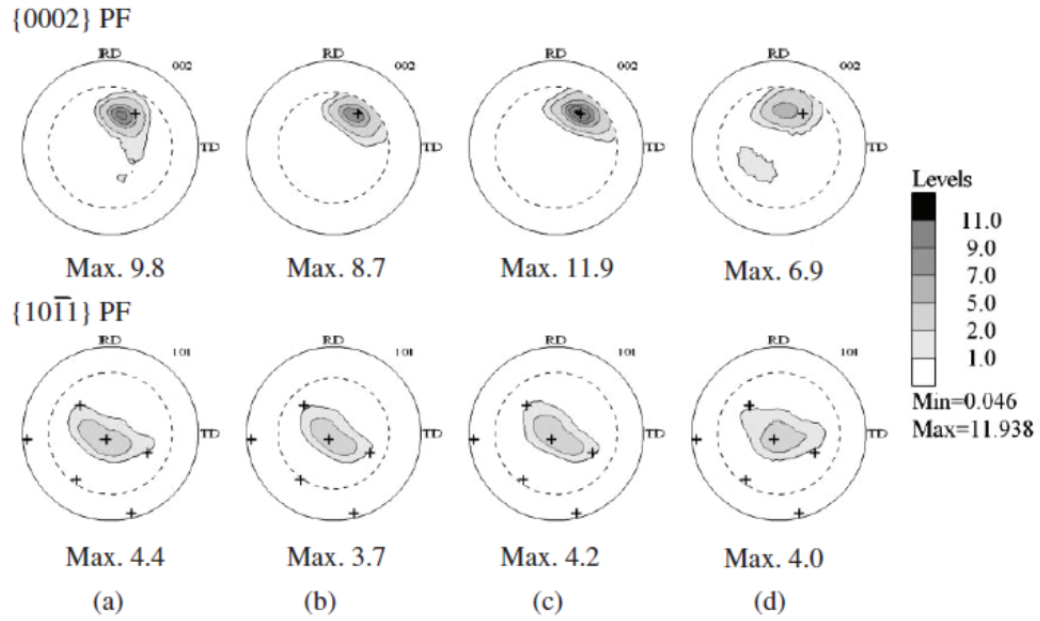


Figure 2-26 The  $\{0002\}$  and  $\{10\bar{1}1\}$  pole figures of the materials after 6 passes at (a) 553 K, (b) 523 K, (c) 493 K, and (d) the material ECAPed at 523K and then annealed at 573K for 1.2 ks. (The  $\{0002\}$  and  $\{10\bar{1}1\}$  poles of  $\{02\bar{2}3\} < \bar{4}154 >$  orientation are marked on the corresponding pole figures using the '+' symbol). [106]



The activation of basal slip increase with the increase in pass number, which results to the significant decrease of yield strength considering the lowest CRSS value of basal slip for Mg in all possible deformation modes at room temperature. The soften effect due to the texture evolution overwhelms the grain size hardening during the ECAP, which leads to the “abnormal” inverse H-P relation. Moreover, when the texture does not change obviously for the samples with 6 pass ECAP at different temperatures, the normal H-P relation are shown clearly. The low grain size dependency is because of the same reason that shows in FSWed samples mentioned above. The easy activation of basal slip of the 6 pass ECAPed samples leads to the small strength coefficient.

#### 2.4.2.3 The influence of deformation modes on the H-P parameters

The potent effect of grain size on the mechanical properties of magnesium alloys has been established in the literature [45, 61, 95, 96, 99-117, 119-121]. In particular, some researchers [103, 122] believe that grain size dependences of mechanical twinning and dislocation glide systems are distinct. Barnett [103] analyzed the H-P parameters for extrusion Mg alloys at the temperature range from room temperature to 200°C (Figure 2-27). The compression samples are loaded along the extrusion direction, which means the loading direction is almost perpendicular to the *c*-axis of crystal considering the strong fiber texture in Mg extrusion bar. Therefore, the extension twin is the dominated deformation mode at room temperature. However, the activation of prismatic slip rises rapidly with the increase in temperature. Therefore, the prismatic slip becomes dominated at higher temperature and smaller grain size.

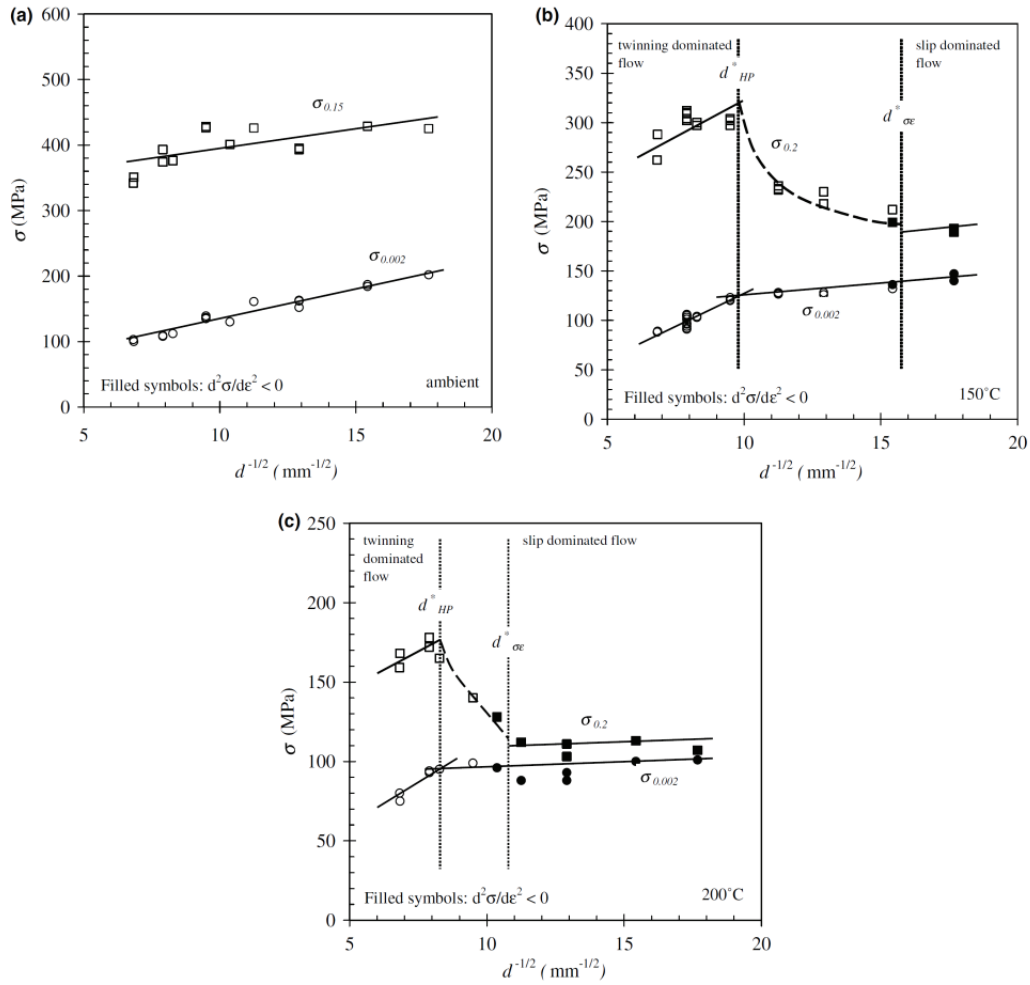


Figure 2-27 H-P plots showing the influence of grain size on  $\sigma_{0.002}$  and  $\sigma_{0.2}$  for: (a) ambient temperature tests, (b) 150 °C and (c) 200 °C. Lines are shown to guide the eye. Twinning-dominated behavior gives way to slip dominated flow at higher temperatures and finer grain sizes [103].

The H-P relationship indicated the transition of deformation modes at 150 °C and 200 °C as well. A “inverse” H-P effect was observed in the study for the stress at a strain of 0.2 for grain sizes between  $d_{HP}^*$  and  $d_{\sigma\epsilon}^*$ . As the grain size decreases in this region, twinning is expected to become increasingly suppressed; i.e., it occurs only in a diminishing number of grains. This suppression of twinning means less twin introduced hardening effects. Therefore, yield stress drops with decrease in grain size. Also, it is revealed that the yield stress follows the H-P relationship for slip dominated flow. It can therefore be surmised that the yield stress is not dependent upon twinning in this region, rather, twinning becomes more significant only after some finite amount of strain. Moreover, the higher grain size dependency of twin than slips modes is indicated. The relationship of H-P parameters,  $\sigma_o$  and  $k_y$  as a function of Zener-Hollomon parameter,  $Z$ , are also obtained from the analysis.

There is precedent for the hypothesis that grain size strengthening affects different deformation mechanisms distinctly. For instance, Ashby [123] proposed that the grain boundary region would serve to accommodate the plastic incompatibilities between distinctly oriented neighbor grains by so-called geometrically necessary dislocations. This model allows the grain interiors to deform via soft (either by virtue of orientation or CRSS) mechanisms while the grain boundary region might require hard mechanisms to operate. This notion is supported by metallographic evidence presented by Hauser [124], who noted non-basal slip throughout the grains in a Mg–Li alloy, but only near the grain boundaries in polycrystalline pure Mg. More recently, Koike [51] published the same kind of observation by TEM of alloy AZ31B, in which it was suggested that non-basal

slip was primarily associated with the grain boundary region. The implication is that hard mechanisms might have more potent grain size dependence than soft mechanisms, which would cause the hard mechanisms to play a more significant role in strain accommodation within fine-grained materials than they do in coarse-grained.

However, this conclusion is argued by Armstrong [125] in his study of another hexagonal close-packed metal, beryllium, in which he also alludes to magnesium. He found that the H-P parameters obtained from tensile flow curves essentially represent the grain size dependence of the critical stress to activate prismatic slip. This is consistent with the notion that prismatic slip is the rate controlling mechanism [126, 127] and recent studies of the present group [128] using neutron diffraction which demonstrate that the activation of prismatic slip is correlated with macroscopic yielding. As such, it seems impossible to reveal the distinct grain size dependences of the different deformation through the H-P slope alone.

Jain [89] measured the H-P parameters of in-plane tension along TD and RD cut from a rolled AZ31 plate at room temperature, which is shown in Figure 2-28. The prismatic slip dominates the deformation for both cases and very few extension twins and contraction twins are involved, which is demonstrated through VPSC modeling. In order to simulated H-P plots, the slip parameters are changed independently, which can illustrate their respective influences on the strength (Figure 2-29). For example, the H-P Law is first used to describe grain size dependence of the basal slip systems,  $\tau_{basal,0}$ , alone and the resulting polycrystal simulations show hardly any grain size effect.

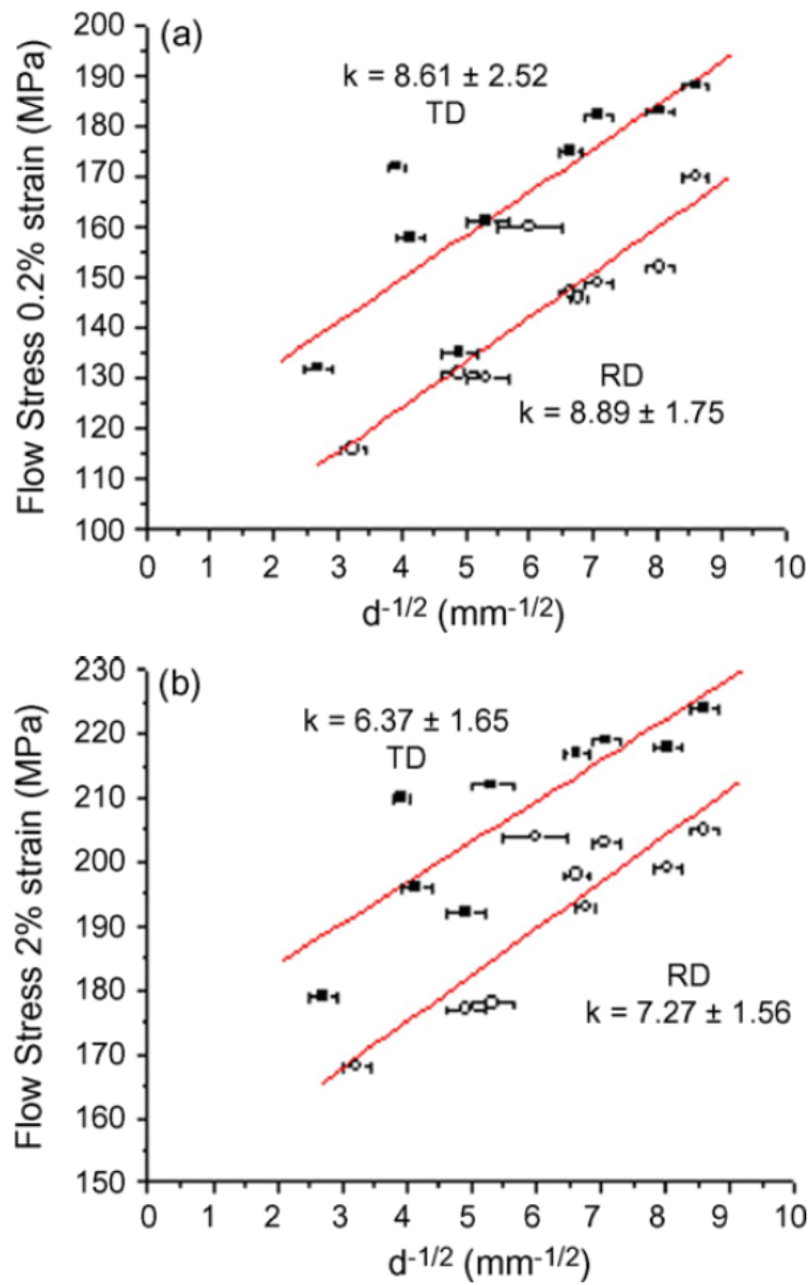


Figure 2-28 H-P plots of the flow stress at (a) 0.2% and (b) 2.0% offset strains [89]

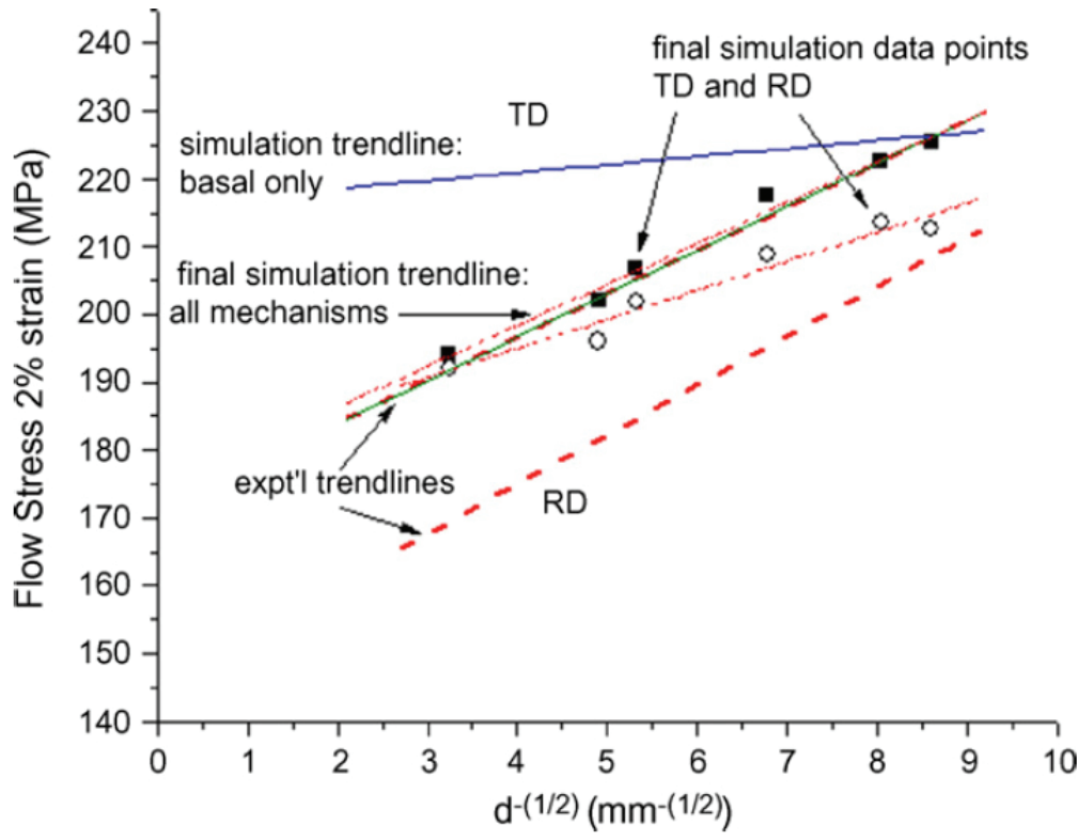


Figure 2-29 Comparison of experimentally determined and simulated H-P trend lines obtained using various assumptions described in the text [89].

In contrast, if the strength of the prismatic slip systems,  $\tau_{\text{prismatic},0}$ , are varied accordingly, the simulated results closely approximate the experimental observation. These state-of-the-art polycrystal plasticity simulation results provide verification (and quantification) of the claim made by Armstrong [125, 129], that the grain size dependence of the strength of prismatic slip controls the grain size dependence of the polycrystal strength of magnesium (and beryllium). Finally, if all the slip modes (and extension twinning) are assumed to have the same type of H-P effect, the results also closely model those of the experiment. Note that only the trend lines resulting from simulations of tension along the TD are shown for the first two cases, for clarity, while each individual simulation result for tension along the TD and RD are shown for the final case, along with their respective linear fits. The variations of the simulated points from the trend lines are due to the use of the experimentally measured textures of the respective grain sizes as modeling inputs.

Based on the VPSC modeling, not only extension twinning was predicted successfully, the observed increase in twin volume fraction (TVF) with increasing grain size is also predicted, when the initial texture is taken into account (Figure 2-30). Interestingly, it is not necessary to change the single crystal constitutive parameters in order to obtain this result, which indicates that the main reason for the increase in twinning activity within larger grain size samples is related to the change in texture and not due to grain size itself.

The VPSC analysis suggests that the grain size dependence of extension twinning is the same as that of the slip mechanisms.

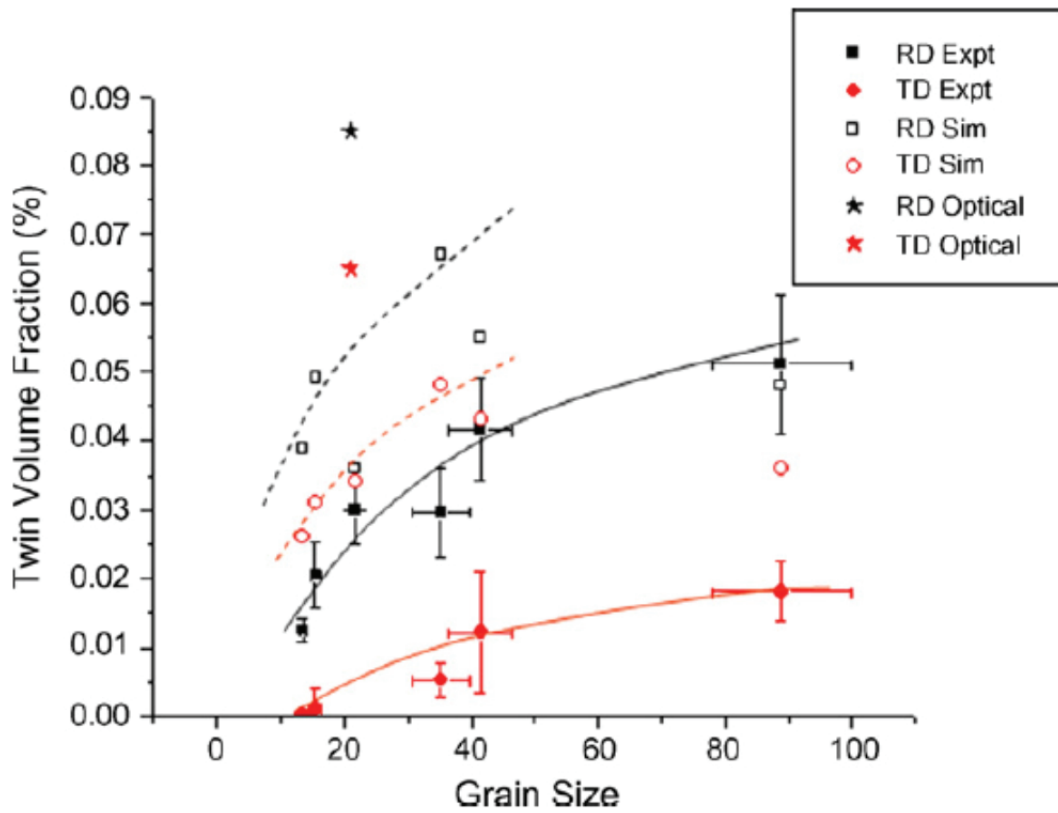


Figure 2-30 Grain size dependence of the twin volume fraction as measured from XRD pole figures shows increasing twinning with increasing grain size. Also shown is the TVF measured from optical micrographs for 21 $\mu$ m grain size samples (stars) and simulated TVF values (open symbols and dashed curves) [89].



If twinning is assumed to have a stronger grain size dependence than slip (as observed previously by Barnett [103]), the twin volume fraction is over-predicted at large grain sizes [89]. An explanation for this apparent contradiction is found in the internal stress measurements by in situ neutron diffraction, which shows that basal slip strongly precedes extension twinning during the present tensile deformation, and the VPSC modeling which shows that slip continues to dominate the strain accommodation throughout the deformation [89]. It is suggested that stress concentrations associated with grain incompatibilities (e.g., dislocation pile-ups) are responsible for activating twinning and, thus, the grain size dependence of slip controls the grain size dependence of twinning. In contrast, extension twinning was the dominant straining mechanism responsible for yielding during the compression tests of extruded Mg alloy, AZ31B, performed by Barnett [102], which allowed the grain size dependence of twinning to be assessed more independently without such a strong influence of dislocation slip as a precursor to twinning.

Thus, the effects of the texture and different deformation modes including the, basal slip, prismatic slip, and extension twin, which are the possible deformation mechanisms at room temperature on the grain size sensitivity of yield strength and texture evolution, are still controversy, which worth to be deeply studied.

## 2.5 The visco-plastic self-consistent modeling

### 2.5.1 *Introduction of self-consistent model*

The anisotropic mechanical behavior of the Mg alloy is well known. Various approaches have been proposed to model the deformation mechanisms and texture evolutions by simulating the crystal reorientation and hardening behaviors based on the deformation mechanisms started from the original Taylor-Bishop-Hill [130] and Sachs [131] approaches to the VPSC theory [132], the  $\phi$ -model [133] and finite element model [134]. The difference of the crystal plasticity models comes from the methods used to describe the interaction between the grains and their neighbors. Actually, the Taylor and Sachs model assume the iso-strain and iso-stress between the grains, respectively, and ignore the interaction between the grains and surroundings. The VPSC simulation assume the surroundings as a homogenous effective medium (HEM). The interaction between each grain (represented by an ellipsoidal shape inclusion) with the HEM can be simplified to various interaction equations such as Tangent and Scent according the Eshelby theory. The  $\phi$ -model is a non-linear intermediate model, which can describe the interaction between the grain and neighbors in the way similar to the VPSC model, while the Eshelby-tensor is not embedded into the model.

In the past about two decades, the anisotropic behaviors and the texture evolution during the deformation of the HCP metals such as Ti, Mg and Zr [135] have been intensively studied through the polycrystal plasticity theory. The pioneering works by Lebensohn [132] led to the VPSC modeling. The micromechanical self-consistent method have the advantages by comparing with the Taylor's and Sach's assumption due

to the much more accurate stress and texture evolution prediction as well as the finite element methods due to lower computational costs.

However, the VPSC models cannot be used to simulate the elastic deformation component or internal stress evolution. Therefore, the elasto-plastic self-consistent (EPSC) polycrystal model was developed by Turner [136] to understand the elastic deformation and internal stress. The EPSC model was well established and verified by comparing the lattice strain measurements through neutron diffraction measurements of HCP crystals such as Mg alloys during the deformation [50, 137]. However, EPSC cannot be used to catch the crystal reorientation, which leads to the texture evolution or twin activation. Moreover, the EPSC model is strain rate-insensitive, and therefore it avoids to simulate the stress relaxations and creep behaviors, while they occur commonly during the long time in-situ neutron diffraction measurements. Very recently, the elasto-viscoplastic self-consistent model (EVPSC) developed by Wang [138] was successfully be used to simulate and explain the lattice strain and texture evolution in the large strain during the uniaxial tension and compression of Mg AZ31 alloy measured through neutron diffraction by Agnew [128]. Overall, the VPSC is the most effective one to understand the deformation mechanisms and especially the texture evolution during the large plastic deformation under different loading paths for the alloys with different structures, since the relatively easier constitutive parameters are needed. For example, it was successfully to simulate the creep texture and creep properties of Al alloy processed by ECAP [139]; texture evolution of Al [140] and Cu [141] after the ECAE; texture gradients in the rolled Al sheet [142]; stress-strain in the ferrite and martensite during the loading of a dual-

phase steel [143], and the twinning behaviors during the compression of the clock-rolled Zr [144].

## 2.5.2 The theory of VPSC model

### 2.5.2.1 Crystal plasticity theory

Plastic deformation of a crystal is assumed to be Voce hardening due to the crystallographic slip and twinning on slip and twinning systems  $(\mathbf{s}^\alpha, \mathbf{n}^\alpha)$ . Here,  $\mathbf{s}^\alpha$  and  $\mathbf{n}^\alpha$  are, respectively, the slip/twinning direction and normal direction of the slip/twinning system  $\alpha$  in the present configuration. The following equation gives the grain (crystal) level plastic strain rate  $\mathbf{d}^g$ ,

$$\mathbf{d}^g = \sum_{\alpha} \dot{\gamma}^{\alpha} \mathbf{P}^{\alpha} \quad 2-9$$

where  $\dot{\gamma}^{\alpha}$  is the shear rate of slip (twinning) system  $\alpha$ , and  $\mathbf{P}^{\alpha}$  is the associated Schmid tensor:

$$\mathbf{P}^{\alpha} = \frac{1}{2} (\mathbf{s}^{\alpha} \mathbf{n}^{\alpha} + \mathbf{n}^{\alpha} \mathbf{s}^{\alpha}) \quad 2-10$$

For slip,

$$\dot{\gamma}^{\alpha} = \dot{\gamma}_0 \left| \frac{\tau^{\alpha}}{\tau_{CR}^{\alpha}} \right|^{1/m} \text{sgn}(\tau^{\alpha}) \quad 2-11$$

and for twinning,

$$\dot{\gamma}^{\alpha} = \begin{cases} \dot{\gamma}_0 \left| \frac{\tau^{\alpha}}{\tau_{CR}^{\alpha}} \right|^{1/m} & \text{for } \tau^{\alpha} > 0 \\ 0 & \text{for } \tau^{\alpha} \leq 0 \end{cases} \quad 2-12$$

where  $\dot{\gamma}_0$  is a reference value of slip/twinning rate,  $m$  is the slip/ twinning rate sensitivity, and  $\tau^\alpha$  is the resolved shear stress:

$$\tau^\alpha = \boldsymbol{\sigma}^g : \mathbf{P}^\alpha \quad 2-13$$

$\tau_{CR}^\alpha$  is the critical resolved shear stress (CRSS), sgn is the sign function.

The evolution of  $\tau_{CR}^\alpha$  is taken in the form of

$$\dot{\tau}_{CR}^\alpha = \frac{d\hat{\tau}^\alpha}{d\gamma_{ac}} \sum_{\beta} h^{\alpha\beta} \dot{\gamma}^\beta \quad 2-14$$

where  $\gamma_{ac} = \sum_{\alpha} |\gamma^\alpha|$  is the accumulated shear strain in the grain,  $h^{\alpha\beta}$  is the latent hardening coupling coefficient which empirically accounts for the obstacles on system  $\alpha$  associated with system  $\beta$ .  $\hat{\tau}^\alpha$  is the threshold stress and is characterized by

$$\hat{\tau}^\alpha = \tau_0^\alpha + (\tau_1^\alpha + h_1^\alpha \gamma_{ac}) (1 - \exp(-\frac{h_0^\alpha}{\tau_1^\alpha} \gamma_{ac})) \quad 2-15$$

Here,  $\tau_0^\alpha$ ,  $h_0^\alpha$ ,  $h_1^\alpha$ , and  $\tau_0^\alpha + \tau_1^\alpha$  are the initial CRSS, the initial hardening rate, the asymptotic hardening rate and the back-extrapolated CRSS, respectively for the deformation mode,  $\alpha$ . These laws regard grains as composite inclusions consisting of parent grain and twin bands and are not considered here.

#### 2.5.2.2 Predominant Twin Reorientation Scheme

The Predominant Twin Reorientation (PTR) scheme proposed by Tome [135] is used in the present chapter to model the reorientation by twinning. Within each grain  $g$ , the PTR scheme tracks the shear strain  $\gamma^{\alpha,g}$  contributed by each twinning system  $\alpha$ , and the associated volume fraction  $V^{\alpha,g} = \gamma^{\alpha,g} / \gamma^{tw}$  as well. Here  $\gamma^{tw}$  is the characteristic

shear (constant) associated with twinning. Crystallographically equivalent twins belong to the same twin mode. For example, the  $\{10\bar{1}2\}$  twinning systems, which are activated by  $c$ -axis tension constitute the tensile twin mode. The sum over all twin systems associated with a given twin mode, and then over all the grains, represents the “accumulated twin fraction”,  $V^{acc,mode}$  in the aggregate for the particular twin mode:

$$V^{acc,mode} = \sum_g \sum_{\alpha} V^{\alpha,g} \quad 2-16$$

In the PTR scheme, after each deformation increment, a grain is randomly selected to check if the predominant twinning system exceeds a threshold value. If so, the grain is entirely reoriented to a new orientation according to predominant twinning system. The volume fraction of this reoriented grain is added to the so called “effective twin fraction”,  $V^{eff,mode}$ . The process is repeated until either all grains are checked or the effective twin fraction exceeds the accumulated twin fraction. In the latter case, the reorientation by twinning is ceased and the next deformation step is considered. The aforementioned threshold value is defined as

$$V^{th,mode} = A^{th1} + A^{th2} \frac{V^{eff,mode}}{V^{acc,mode}} \quad 2-17$$

,where  $A^{th1}$  and  $A^{th2}$  are two material constants. This approach statistically solves the practical problem that the number of orientations would grow continually if each activated twinning system was represented by a new orientation. Additionally, it maintains the twinned volume fraction at a level that is consistent with the shear activity of the twins contributing to the deformation.

### 2.5.2.3 Self-consistent homogenizing methods

The Eshelby inclusion formalism, which assumes each grain as an ellipsoidal inclusion embedded in a HEM is used to describe the interaction between each grain and the neighbor. During the each deformation step, the single crystal constitutive behavior, which describes the grain-level response, and the self-consistency criteria are solved simultaneously, which makes the grain-level stresses and strain rates to be consistent with the boundary conditions imposed on the surrounding polycrystalline aggregate.

Different approximation methods are used to linearize the visco-plastic response. The linearized behavior of an inclusion (single crystal) can be written as

$$\mathbf{d}^g = \mathbf{M}^g : \boldsymbol{\sigma}^g + \mathbf{d}_0^g \quad 2-18$$

, where  $\mathbf{M}^g$  and  $\mathbf{d}_0^g$  are the visco-plastic compliance and the back-extrapolated term of grain,  $g$ , respectively. The linearization of HEM (polycrystal) is analogous to the inclusion and is written as:

$$\mathbf{D} = \bar{\mathbf{M}} : \boldsymbol{\Sigma} + \mathbf{D}_0 \quad 2-19$$

, where  $\bar{\mathbf{M}}$  and  $\boldsymbol{\Sigma}$  are the visco-plastic compliance and back-extrapolated term of the HEM, respectively.

Different stiffness of grain-matrix interaction associated with the various self-consistent schemes (SCS) can be chosen for the linearization. The first one is the Secant SCS:

$$M_{ijkl}^{g,scant} = \gamma_o \sum_{\alpha} \left( \frac{\tau^{\alpha}}{\tau_{cr}^{\alpha}} \right)^{\frac{1}{m}-1} \frac{P_{ij}^{\alpha} P_{kl}^{\alpha}}{\tau_{cr}^{\alpha}} \quad 2-20$$

$$d_{0ij}^{g,scnt} = 0 \quad 2-21$$

The affine SCS linearization is

$$M_{ijkl}^{g,affine} = \frac{\dot{\gamma}_o}{m} \sum_{\alpha} \left( \frac{\tau^{\alpha}}{\tau_{cr}^{\alpha}} \right)^{\frac{1}{m}-1} \frac{P_{ij}^{\alpha} P_{kl}^{\alpha}}{\tau_{cr}^{\alpha}} \quad 2-22$$

$$d_{0ij}^{g,affine} = \left( 1 - \frac{1}{m} \right) d_{ij}^g \quad 2-23$$

The relation of grain-level stress and strain rate to the aggregate response is obtained self-consistently by

$$(\mathbf{d}^g - \mathbf{D}) = -\tilde{\mathbf{M}} : (\boldsymbol{\sigma}^g - \boldsymbol{\Sigma}) \quad 2-24$$

where the interaction tensor  $\tilde{\mathbf{M}}$  is given by:

$$\tilde{\mathbf{M}} = (\mathbf{I} - \mathbf{S}^{-1}) : \mathbf{S} : \bar{\mathbf{M}} \quad 2-25$$

, where  $\mathbf{S}$  is the Eshelby tensor for a given grain,  $\mathbf{I}$  is the identity tensor. The condition that  $\mathbf{D} = \langle \mathbf{d}^g \rangle$  and  $\boldsymbol{\Sigma} = \langle \boldsymbol{\sigma}^g \rangle$  (where  $\langle \dots \rangle$  represents the volume average) leads to a self-consistent equation giving the macroscopic compliance as:  $\bar{\mathbf{M}} = \langle \mathbf{M}^g : \mathbf{B}^g \rangle : \langle \mathbf{B}^g \rangle^{-1}$

$$2-26$$

and

$$\mathbf{B}^g = (\mathbf{M}^g + \tilde{\mathbf{M}}) : (\bar{\mathbf{M}} + \tilde{\mathbf{M}})^{-1} \quad 2-27$$

Therefore, the interaction tension is approximately proportional to the macroscopic viscoplastic compliance, which is itself given by a weighted average of the vico-plastic compliances of the grains. The macroscopic compliance and the single grain compliances are of the same order of magnitude, since the macroscopic compliance is a weighted average of the single grain.



The Tangent SCS is given by

$$\tilde{\mathbf{M}} = \frac{1}{m}(\mathbf{I} - \mathbf{S}^{-1}) : \mathbf{S} : \bar{\mathbf{M}}^{secant} \quad 2-28$$

Therefore, the Secant and Tangent SCS represent the upper and lower boundaries of the interaction, respectively, and the  $m^{eff}$  SCS provides an intermediate interaction tensor as:

$$\tilde{\mathbf{M}} = \frac{1}{m^{eff}}(\mathbf{I} - \mathbf{S}^{-1}) : \mathbf{S} : \bar{\mathbf{M}}^{secant} \quad 2-29$$

where  $m < m^{eff} < 1$ .

The assessment of the predictive capability of the VPSC model with various SCSs was studied by H. Wang [145]. The material parameters for the various models were first fitted to experimental uniaxial tension and compression curves along the RD (shown in Figure 2-31) and then used to predict the uniaxial tension and compression along TD (Figure 2-32) and uniaxial compression along ND (Figure 2-33). Therefore, the predict capability is represented by the difference between the predicted stress response and R values, which is defined as transvers-to-thickness plastic strain-ratio under uniaxial tension within the sheet plane. It was found that the predictive capability of Secant scheme and classic Taylor model are almost the same for all the deformation processes. Moreover, the Affine scheme provides the best overall prediction results with less artificial tuning by comparing with the  $m^{eff}$  scheme. The Tangent model slightly underestimated the flow stress when the strain is less than 0.005 and overestimated the hardening at large strain for the compression along ND, while it provided the best prediction of the R value for this loading path, which is shown in (Figure 2-34).

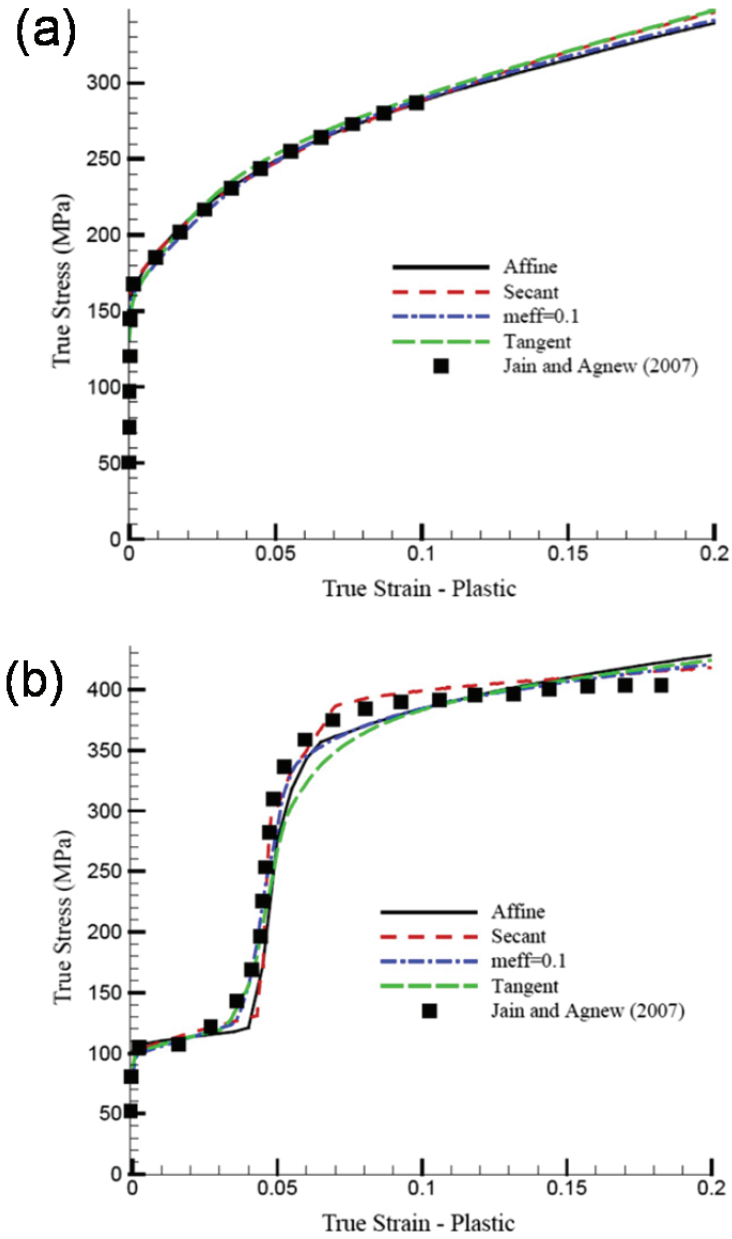


Figure 2-31 Stress-strain curves under uniaxial tension and compression along the RD:

(a) uniaxial tension, and (b) uniaxial compression [145]

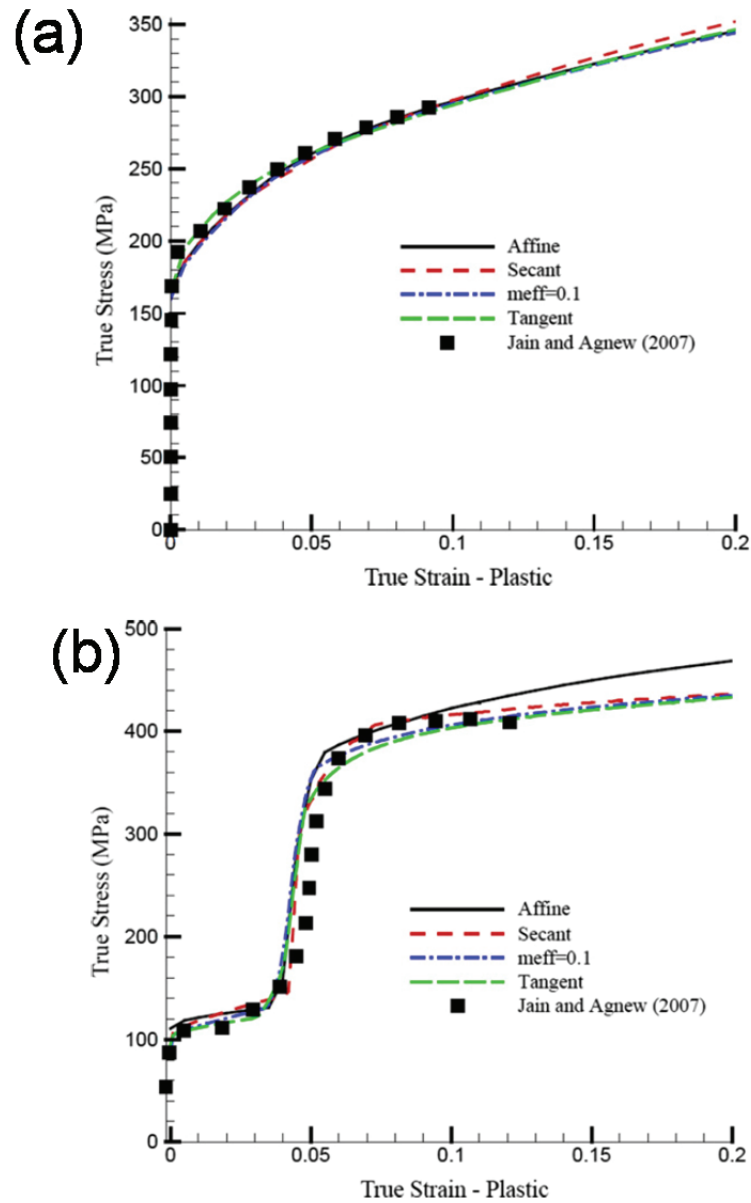


Figure 2-32 Stress-strain curves under uniaxial tension and compression along the TD obtained through experiment (symbols) and VPSC simulation with different SCS (solid lines): (a) uniaxial tension, and (b) uniaxial compression [145]

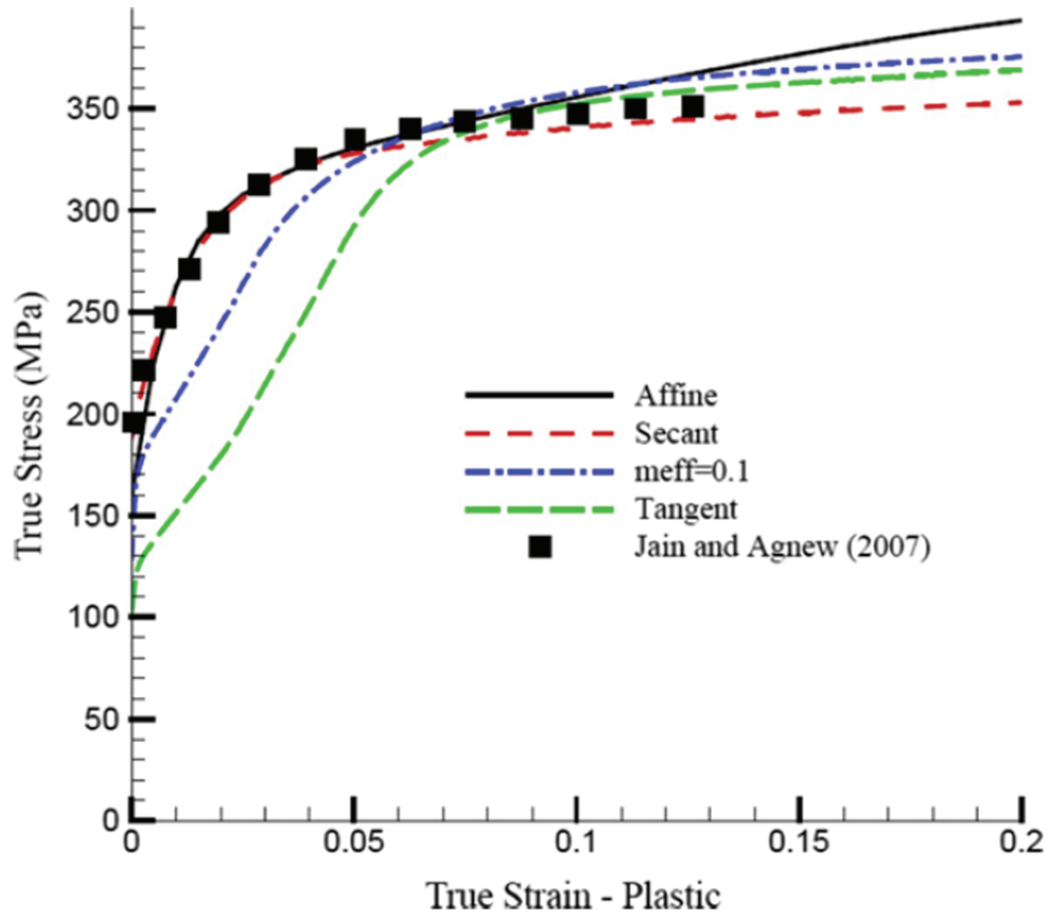


Figure 2-33 Stress-strain curves uniaxial compression along the ND obtained through experiment (symbols) and VPSC simulation with different SCS (solid lines) [145]

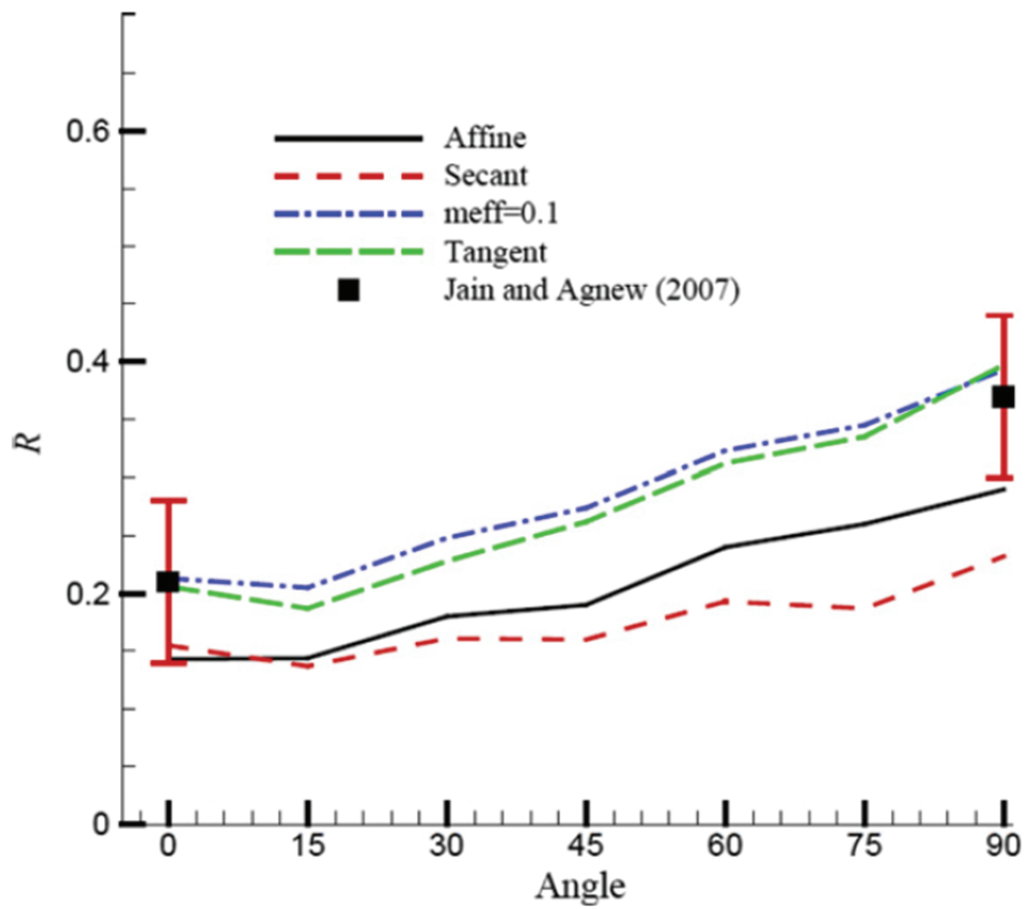


Figure 2-34 R value under uniaxial compression with the sheet plane obtained through experiment (symbols) and VPSC simulation with different SCS (solid lines) [145]

### ***2.5.3 The VPSC simulation of texture evolution and activation of deformation modes for Mg alloys***

Based on the VPSC theory mentioned above (Voce hardening and PTR scheme), the deformation mode activation, strain-stress curves and texture evolution during the deformation can be modeled. A lot of research results [29, 65, 71, 72, 86, 89, 97, 146] demonstrated that VPSC modeling can explicate the deformation mechanism and texture evolution accurately for wide range of deformation temperature. For example, Jain [35] proved the accurate VPSC modeling of Mg compression deformation behavior from the range of room temperature to 250°C of a rolled Mg plate (Figure 2-35). Four different deformation modes are considered in the modeling including basal, prismatic and  $\langle c+a \rangle$  pyramidal slip as well as extension twin. The best fit input parameters for different deformation temperature they used for the modeling are shown in Table 2-8.

The yield strength during in-plane compression (IPC) is insensitive to temperature. Thus, the CRSS values of the athermal mechanisms of basal slip and tensile twinning are held essentially constant. In fact, it is necessary to raise the CRSS for twinning slightly to correctly predict the observed slight increase in flow stress, as well as the decrease in twin volume fraction observed at 200 °C. However, the parameters describing the thermally activated prismatic  $\langle a \rangle$  and pyramidal  $\langle c+a \rangle$  slip modes are softened with increasing temperature.[35]

The polycrystal modeling technique allows for an indirect assessment of the grain-level plastic deformation mechanisms in terms of their CRSS,  $\tau_0^\alpha$ , and back-extrapolated flow stress,  $\tau_0^\alpha + \tau_1^\alpha$ , values as a function of temperature (Figure 2-36).

Table 2-8 Best-fit model parameters describing the CRSS and hardening responses of the four deformation mechanisms as a function of temperature. The stress values are normalized by the CRSS for basal slip, which is fixed at ~25MPa for all temperatures [35].

| Temperature | Mode  | $\tau_o$ | $\tau_1$ | $\theta_o$ | $\theta_1$ | Latent |
|-------------|-------|----------|----------|------------|------------|--------|
| 20          | Basal | 1        | 0.5      | 80         | 3          | 4      |
|             | Prism | 3.2      | 2        | 20         | 0.5        | 4      |
|             | <c+a> | 5        | 6        | 500        | 0          | 2      |
|             | Twin  | 1.5      | 0        | 0          | 0          | 4      |
| 150         | Basal | 1        | 0.5      | 80         | 3          | 1.5    |
|             | Prism | 2        | 2.5      | 20         | 0          | 1.5    |
|             | <c+a> | 2        | 5        | 100        | 0          | 1      |
|             | Twin  | 1.7      | 0        | 0          | 0          | 1.5    |
| 175         | Basal | 1        | 0.5      | 80         | 1          | 1.2    |
|             | Prism | 1.9      | 1.5      | 20         | 0          | 1.2    |
|             | <c+a> | 1.9      | 3.3      | 50         | 0          | 1      |
|             | Twin  | 1.9      | 0        | 0          | 0          | 1.2    |
| 200         | Basal | 1        | 0.5      | 80         | 0          | 1.2    |
|             | Prism | 1.8      | 0.8      | 20         | 0          | 1.2    |
|             | <c+a> | 1.8      | 2        | 35         | 0          | 1      |
|             | Twin  | 2.1      | 0        | 0          | 0          | 1.2    |

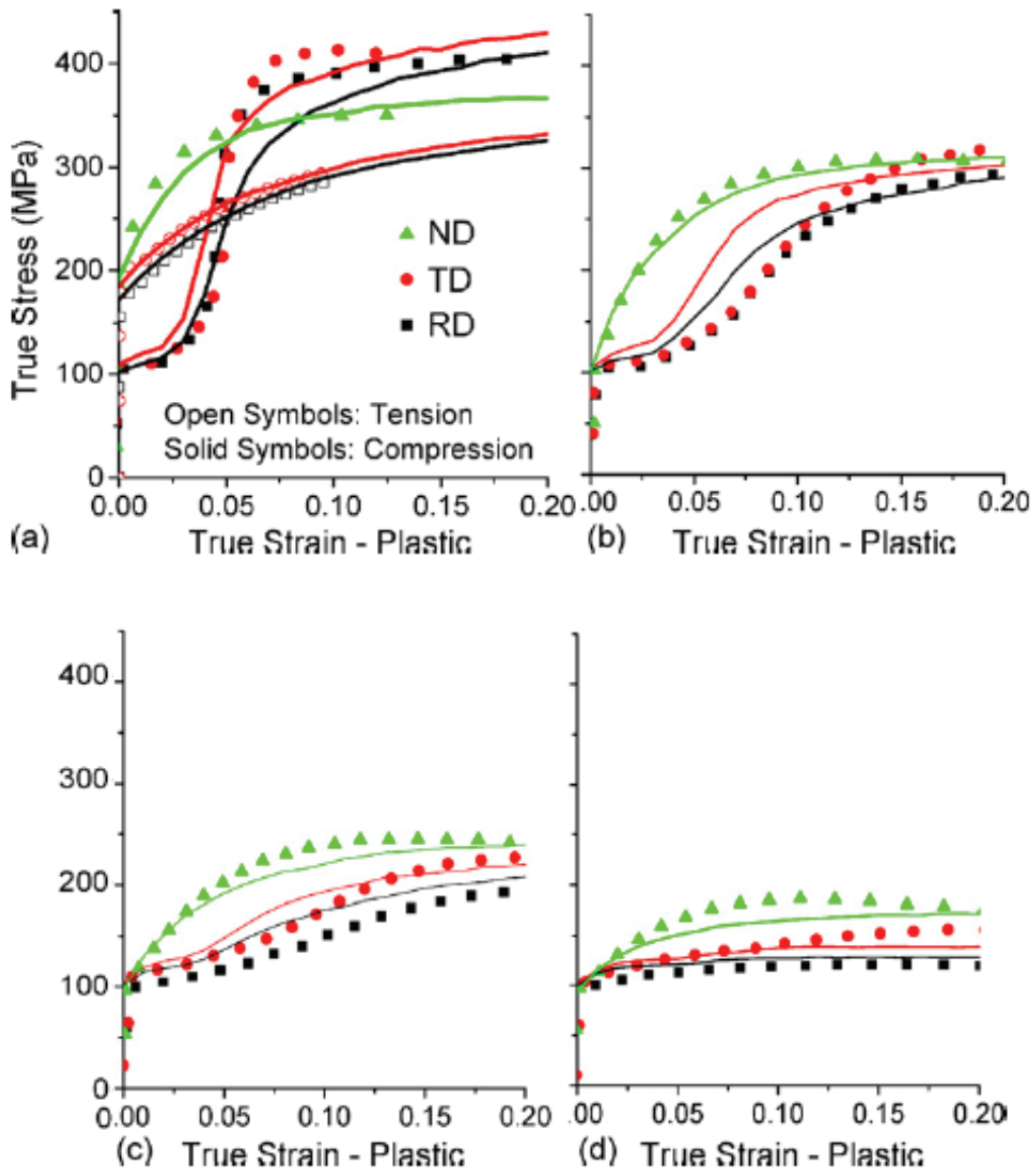


Figure 2-35 A comparison of experimental (symbols) and VPSC simulated (curves) compressive flow behavior at (a) RT, (b) 150 °C , (c) 175 °C and (d) 200 °C [35].



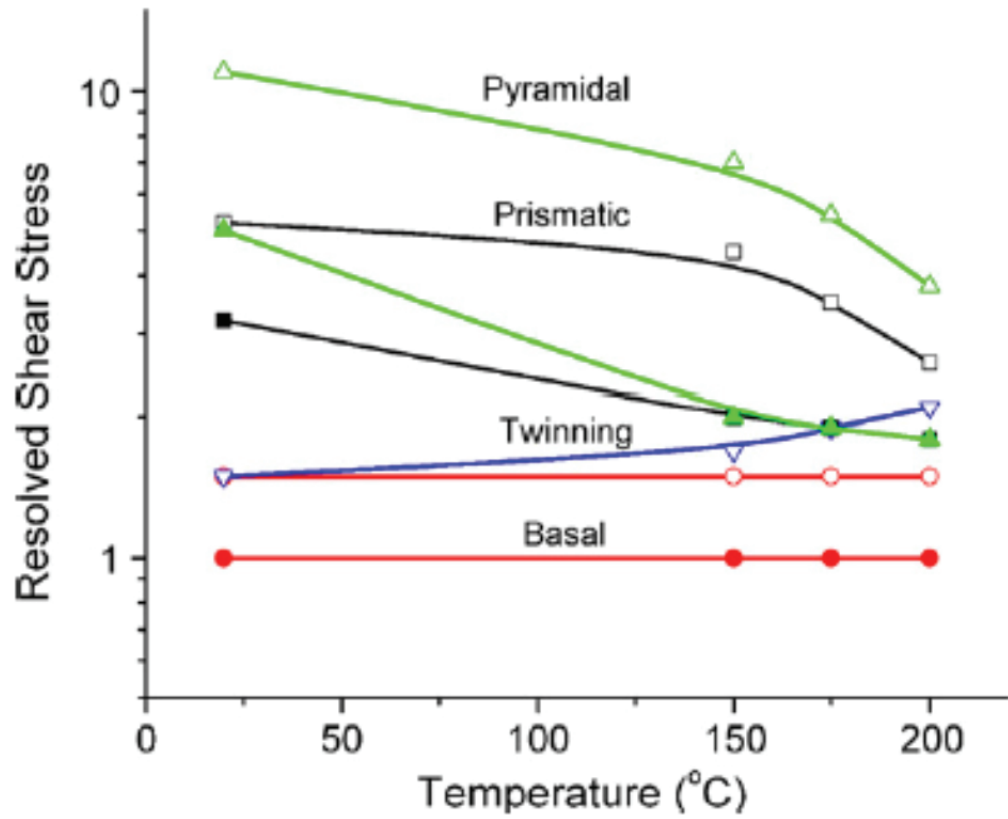


Figure 2-36 Variation of CRSS,  $\tau_0^\alpha$  (solid symbols) and back extrapolated flow stress,  $\tau_0^\alpha + \tau_1^\alpha$  (open symbols) for different deformation modes as a function of temperature. The stresses are normalized by the CRSS for basal slip.[35]

In the aforementioned single crystal studies, the CRSS ratios of non-basal to basal slip are reported to be of order 40:1–80:1 at room temperature from experiments, while those of the present polycrystal model are of order 2:1–5:1 (i.e., an order of magnitude lower). This difference in single crystal and polycrystal behavior may be related to the grain boundaries acting as potent sources and sinks for dislocations or due to grain boundary constraint. Interestingly, despite the huge difference in the magnitude of these ratios, the relative decrease in the CRSS values of thermally activated non-basal  $\langle a \rangle$  and  $\langle c+a \rangle$  slip mechanisms is similar to that reported for single crystals over the observed temperature range. At these moderate homologous temperatures, basal slip is observed to behave athermally, such that further increase in temperature has no marked effect on the critical stress for activation, also in agreement with single crystal studies.

The latent hard parameters of modeling are relatively tricky. Grain size refinement is an effective strengthening mechanism and twin boundaries are a special class of grain boundaries. As such, it is not surprising that twinning leads to a measurable level of latent hardening. It has already been mentioned that the terminal flow stresses are higher after IPC than after through-thickness compression (TTC), which is shown in Figure 2-35. The difference between the two is modeled in an *ad hoc* fashion by including an increased level of latent hardening of the slip modes due to twinning (Table 2-8). In general, the different slip modes are expected to interact with specific twin boundary variants in different ways. For instance, in order to predict the observed saturation of the in-plane compression curves at high strains, a lower latent hardening parameter for  $\langle c+a \rangle$  slip than for the  $\langle a \rangle$  slip modes at room temperature was invoked

[29, 30, 35]. Since twinning and  $\langle c+a \rangle$  slip both occur on pyramidal planes, albeit different ones, the interactions between the two may be weaker than other slip modes which will inevitably be forced to interact. Further, interactions between  $\langle a \rangle$  dislocations and  $\{10\bar{1}2\}$  type twin boundaries can give rise to  $\langle c+a \rangle$  dislocations, thereby promoting the mechanism. Finally, as the temperature is raised, the latent hardening parameters must be decreased to correctly predict the rapidly decreasing flow stresses, and this reflects the notion that the dislocation–twin boundary interactions (or required recovery mechanisms) are thermally activated.

#### ***2.5.4 Further Development of VPSC model***

##### **2.5.4.1 Composite grain (CG) model**

Tome proposed a PTR scheme to describe the activation of twinning and crystal reorientation due to the twinning [135]. The PTR scheme allows one to account for the contribution to texture of every twin system in every grain by transferring volume fraction from the parent to the daughter twin. However, in the PTR model, the grain identity is lost and does not allow one to implement a realistic hardening scheme. In order to catch the hardening caused by the twinning such as the barrier effect of the twin boundaries on the dislocation movement, the latent hardening scheme in the VPSC code was used as mentioned above. It was reported that the stress-strain behaviors and texture evolution can be accurately simulated for the monotonic compression and tension for Zr at room temperature and liquid nitrogen temperature by selecting the suitable latent hardening parameter of extension twin on other slip systems [135]. However, the ultimate

challenge of a constitutive description is to predict the plastic response associated with complex loading histories, such as changes in loading path. In such a case, the latent hardening law overlooks the directionality of the grain microstructure created during the previous deformation path, and a model with a realistic description of the twin structure and the twin-dislocation interaction inside grains is required.

Therefore, Proust [147] proposed a meso-scale composite grain (CG) model, which incorporates microstructure information of twinning into the polycrystal model. The CG model represents an ellipsoidal inclusion formed by alternating layers of twin and matrix domains. The alternating twin-matrix interfaces provide a barrier of dislocation and define a directional mean free path changes for the propagation of twinning, which provide the hardening effect caused by the microstructure changes due to the twinning.

Figure 2-37 shows the illustration of the CG model by selecting one predominant twin system (PTS) in each grain. The lamellae twin is form and evolves with twin activation. Therefore, two parameters are required to describe the microstructure of the twin: the separation distance  $d^c$  of the center of the lamellae and the maximum volume fraction of the grain that may be reoriented by twinning  $f_{max}^{PTS}$ , which prevent further growth of the twin in the grain.

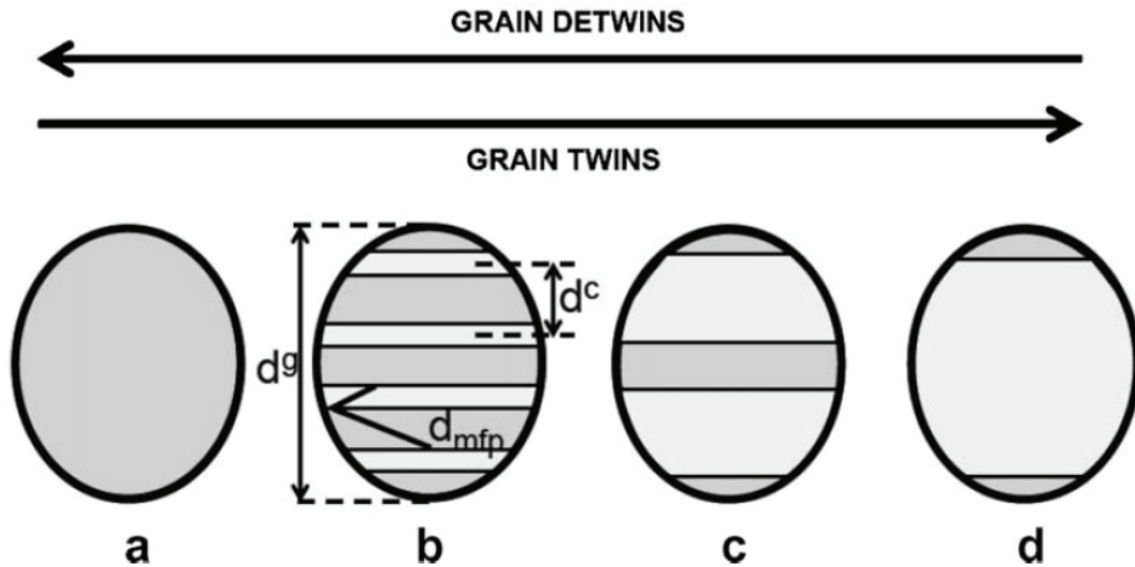


Figure 2-37 Schematics of the CG showing the characteristic lengths used in the model and the evolution of these lengths with the PTS volume fraction when the material twins and detwins. (a) The material has not started to twin and the grain is only constituted of a matrix region, (b) when the material starts to twin, several thin lamellae are created, (c) as the PTS volume fraction increases, some lamellae merge together increasing the mean free  $d_{mfp}^s$  for the dislocation motion inside the twinned domains, and (d) the grain has almost completed twinned and we have now a single twinned region [146]

In the CG model, three factors contribute to the hardening effect: the evolution of the statistical dislocation with the increase in the strain (STAT), which is the classic Voce hardening behavior; the evolution of geometrically necessary dislocation (GND), which is dependent on the directional mean free path  $d_{mfp}^s$  defined as the lamellar spacing of the PTS; and the last one is the Hall-Petch like effect. Therefore, the hardening law can be written as:

$$\tau^s = \tau_{STAT}^s + \tau_{GND}^s + \tau_{HP}^s \quad 2-30$$

where

$$\Delta\tau_{STAT}^s = \frac{d\hat{\tau}^s}{d\Gamma} \sum_{s'} h^{ss'} \Delta\gamma^{s'} \quad 2-31$$

$$\Delta\tau_{GND}^s = \frac{H_{GND}^s}{d_{mfp}^s (\tau_{STAT}^s + \tau_{GND}^s)} \Delta\gamma^s \quad 2-32$$

$$\tau_{HP}^s = \frac{H_{HP}^s}{\sqrt{d_{mfp}^s}} \quad 2-33$$

It should be noted that the twin should have another hardening effect, Basinski hardening, which transfer the dislocation from glissile to sessile as a result of the crystallographic reorientation, while it is not considered in the CG model.

By using the CG model in the VPSC simulation, the stress-strain behaviors of the complex loading, such as the TTC followed by IPC can be simulated to study the interaction between the twin and slip systems (Figure 2-38) [146]. Moreover, it was the first model which allows the detwinning to happen during the complex loading path (Figure 2-38e).

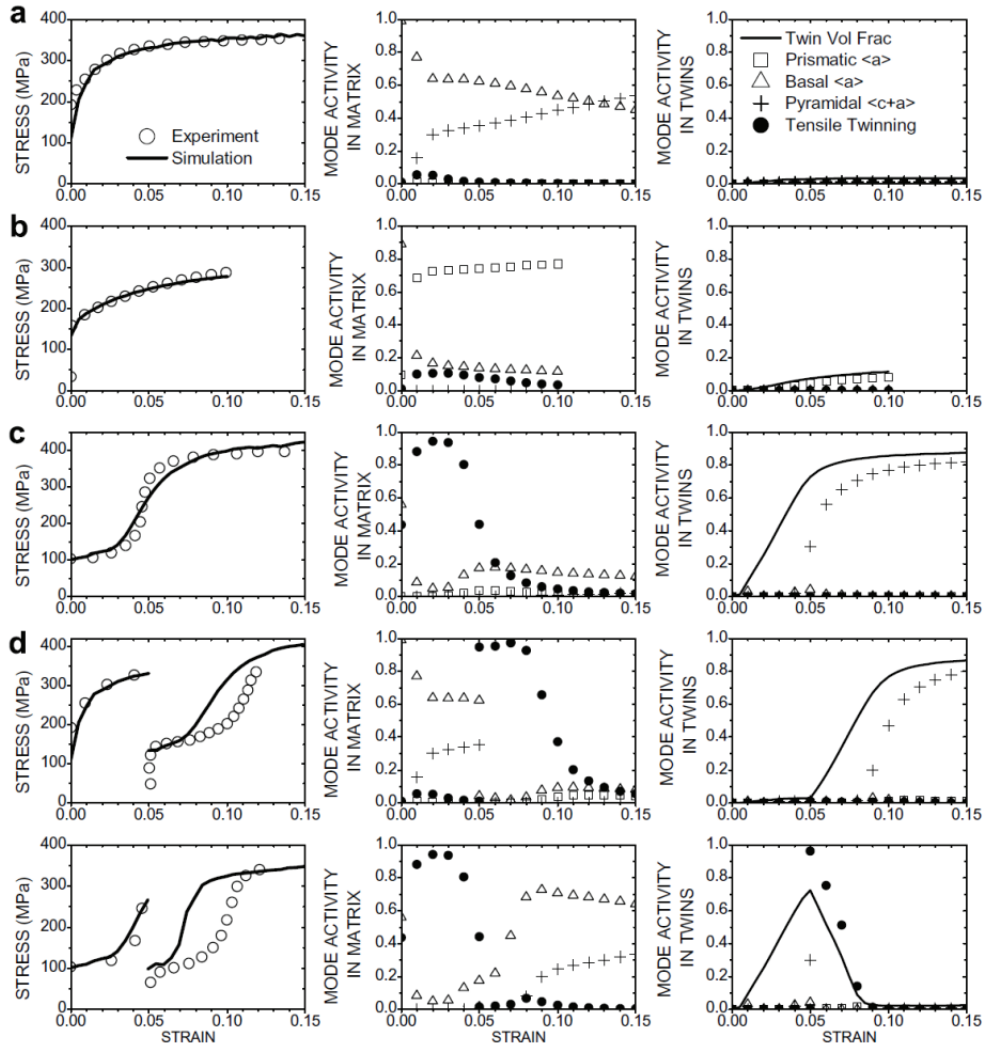


Figure 2-38 Comparison between the experimental (symbols) and predicted (solid lines) stress–strain curves, predicted deformation mode activities in matrix and in twins (the prismatic activity is represented by open squares, the basal activity by open triangles, the pyramidal activity by crosses and the tensile twinning activity by solid circles. The solid line in the activity plot in the twins represents the evolution of the twin volume fraction with strain) for (a) monotonic TTC, (b) monotonic IPT, (c) monotonic IPC, (d) TTC followed by IPC, and (e) IPC followed by TTC. [146]

During the IPC follow by the TTC, the twin transforms back into the matrix orientation and shrinks while the volume fraction of the matrix increases. Finally, the H-P parameters in the model correspond to the barrier effect caused by the extension twin can also be used to predict the monotonic IPT response of the same alloy with different initial grain size, which is shown in (Figure 2-39). Considering the prismatic slip dominates the deformation during the IPC, the H-P parameters of prismatic slip determines the grain size effect on the stress-strain behaviors in this case. It was found that the best parameter was  $100\text{MPa} \cdot \mu\text{m}^{1/2}$ .

#### 2.5.4.2 Dislocation transmutation model

It was mentioned above that the Voce hardening with the in cooperation of PTR scheme of the twinning was used to simulate the deformation mechanisms and texture development of the HCP metals under different loading paths and temperatures. For instance, the mechanical behaviors and texture evolution of Zr deformed at various temperatures ranging from 76 to 300K were successfully predicted [148]. However, the way to describe the twinning is so simplified that some empirical latent hardening parameters which indicates the twin–twin and twin–dislocation interaction are required to tune the results. Moreover, it was very hard to predict the twin activation and deformation behavior under complex loading. For instance, the twinning was introduced through the pre-loading. Therefore, CG model was introduced to solve this issue. However, some parameters specific to a given temperature and strain are required rate in CG model to make it difficult to explain the deformation with various temperature.



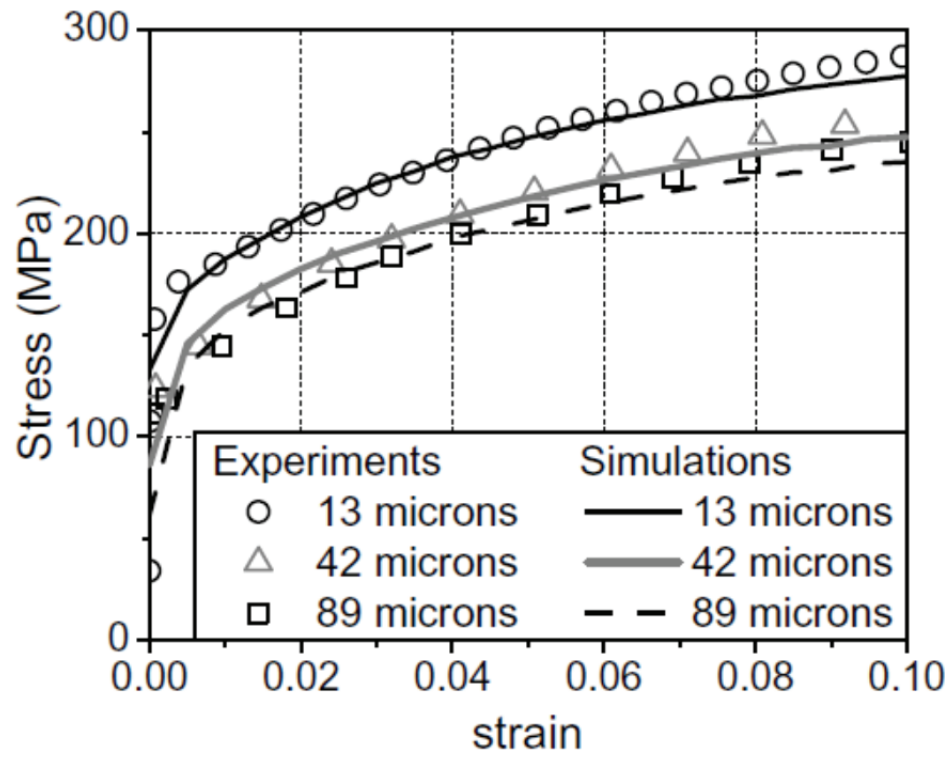


Figure 2-39 Hall–Petch effect due to the initial grain size during IPT. Comparison of the experimental and predicted stress–strain curves. [146]

In this case, the more physical based generalized model is required to break such limitation. In 2008, Beyerlein [149] introduced a dislocation-density-based constitutive rule to describe the deformation of HCP metals with different temperatures instead of the using of the empirical Voce hardening law.

It was known that each slip modes has the dislocation forest and annihilation or debris stage (substructure). If denotes a slip mode having as a slip system, and denotes a twin mode having as a twin system, The brief introduction of the dislocation-density-based hardening law is as following [36]:

$$\tau_c^s = \tau_o^\alpha + \tau_{forest}^\alpha + \tau_{sub}^\alpha + \tau_{HP}^s \quad (for\ slip) \quad 2-34$$

$$\tau_c^t = \tau_o^\beta + \tau_{HP}^t + \tau_{slip}^\beta \quad (for\ twin) \quad 2-35$$

The critical resolve shear stresses for the slip resistance equation take the following forms for each of the forest, substructure and Hall-Petch effects, respectively:

$$\tau_{forest}^\alpha = b^\alpha \chi \mu \sqrt{\rho}^\alpha \quad 2-36$$

$$\tau_{sub}^\alpha = k_{sub} \mu b^\alpha \sqrt{\rho_{sub}} \log \left( \frac{1}{b^\alpha \sqrt{\rho_{sub}}} \right) \quad 2-37$$

$$\tau_{HP}^s = \mu_{HP}^\alpha \sqrt{\frac{b^\alpha}{d_g}} \quad 2-38$$

Here,  $b^\alpha$  is the Burgers vector,  $\chi$  is the dislocation interaction coefficient,  $\mu$  is the shear modulus, and  $d_g$  is the spacing between the twin lamellae.

In order to describe the effect of twinning on the hardening, a fitting parameter twin storage factor (TSF) was introduced to make the twins harden more than the parent

through a higher latent hardening in the twin by repaid increase in the dislocation multiplicity in the twin according to the dislocation transmutation theory.

$$\frac{\partial \rho^\alpha}{\partial \gamma^\alpha} = \frac{\partial \rho_{generation}}{\partial \gamma^\alpha} - \frac{\partial \rho_{removal}}{\partial \gamma^\alpha} = TSF k_1^\alpha \sqrt{\rho^\alpha} - k_2^\alpha(\dot{\epsilon}, T) \rho^\alpha$$

$$\frac{k_2^\alpha(\dot{\epsilon}, T)}{k_1^\alpha} = \frac{b^\alpha \chi}{g^\alpha} \left[ 1 - \frac{kT}{D^\alpha (b^\alpha)^3} \log \left( \frac{\dot{\epsilon}}{\dot{\epsilon}_0} \right) \right] \quad 2-40$$

, where  $D^\alpha$  is the drag stress,  $g$  is the normalized stress-independent activation energy, and  $\dot{\epsilon}$  is the strain rate.

For the twin system,

$$\tau_{HP}^t = \frac{HP^\beta}{\sqrt{d_g}} \quad \text{when } t \text{ is the PTS} \quad 2-41$$

$$\tau_{HP}^t = \frac{HP_{TW}^{\beta\beta'}}{\sqrt{d_{mfp}^s}} \quad \text{when } t \text{ is not the PTS} \quad 2-42$$

$$\tau_{slip}^\beta = \mu \sum_\alpha C^{\beta\alpha} b^\beta b^\alpha \rho^\alpha \quad 2-43$$

, where  $C^{\beta\alpha}$  is the interaction between twin and slip. It was proposed that the interaction of the twin on the hardening can be described by changing the TSF instead of the simplified Hall-Petch like effect from the grain refinement by twinning in the CG model.

It was reported by Oppedal [72] that TSF has to be increased to catch the hardening rate during the IPC of the pure Mg which is shown in Figure 2-40. It was indicated that according to the transmutation theory, the higher hardening in the twins over that in the parent caused by the rapid increase in the dislocation multiplicity within the twin instead of the increase in the dislocation generation.

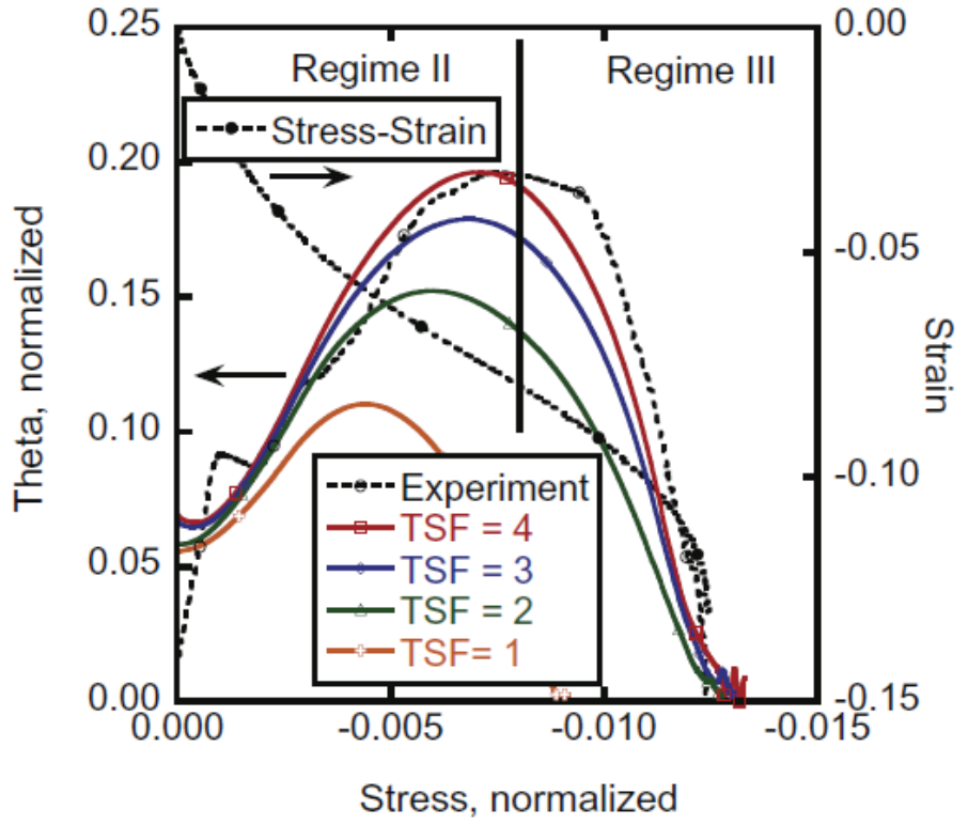


Figure 2-40 Strain hardening rate versus stress plots from compression tests and simulation results with compression twins and four values of twin storage factor, namely unity, two, three and four on a pure magnesium tested along the in-plane direction (IPC) that corresponds to the transverse direction of the sheet. Stress value was normalized as:  $\sigma_{normalized} = (\sigma - \sigma_y)/\mu$ , where  $\sigma_y$  is the yield strength and  $\mu$  is the shear modulus.

## **2.6 The plastic deformation in Mg studied by neutron and synchrotron X-ray Diffraction**

It is mentioned that the input parameters used in the crystal plasticity models such as VPSC, EPSC and EVPSC model need to be verified through the experimental results including stress-strain behaviors, texture evolution, twin volume fraction and lattice strain. Recently, the texture, twin volume fraction and lattice strain can be obtained through the in-situ or ex-situ neutron or synchrotron x-ray diffraction [59, 128, 137, 150, 151]. Therefore, it is found that the diffraction measurement and crystal plasticity simulation are always combined together to study the plastic deformation in Mg alloys. In the following part, some examples including the texture evolution, twinning volume fraction and lattice strain measurements through either S-XRD or neutron diffraction measurement will be presented.

### ***2.6.1 Texture evolution measured by S-XRD***

High energy synchrotron X-rays provide a high penetration depth combined with excellent brilliance, so that texture measurements can be carried out in transmission geometry on bulk samples. This eliminates spurious surface effects often present in texture measurements with conventional laboratory X-ray source or electron backscattering (EBSD) and provides a better statistical average of the microstructure. By employing transmission geometry with an area detector, full Debye – Scherrer rings are recorded simultaneously so that a significant reduction of measuring time is obtained.

This improvement in temporal resolution makes synchrotron X-rays attractive for in situ texture measurements.

Wu [46] used the S-XRD to study the texture evolution during the low-cycle fatigue behavior of a rolled magnesium alloy, AZ31B. The texture evolution is related to the twinning and detwinning during the plastic deformation. The texture evolution during the cyclical loading along RD shows that most grains are initially oriented with their *c*-axes parallel to the ND direction, and the prismatic poles are essentially randomly distributed in the rolling plane. Therefore, the initial grains are preferable for the activation of extension twinning if the alloy is compressed within the plate plane. As mentioned earlier, after the alloy is compressively yielded, the hardening rate is so low that the flow stress keeps almost constant until the maximum compressive strain at -3.0%, where a large volume fraction of grains are reoriented due to extension twinning, resulting in the basal poles aligned with the RD direction (e.g., the loading axis) (Figure 2-41b), and, concurrently, a great number of twin bands are observed in most grains. When the alloy is unloaded to the zero stress at the point “c” on the hysteresis loop in Figure 2-41, no significant change in the pole figures (Figure 2-41c) is observed, compared to Figure 2-41b. At a strain of 0%, the texture is completely reversed (Figure 2-41d) which provides the strong evidence that the twins return to the original orientation by detwinning, rather than retwinning, where the twins are replaced by new twin variants, which would result in a deviation from the initial texture.

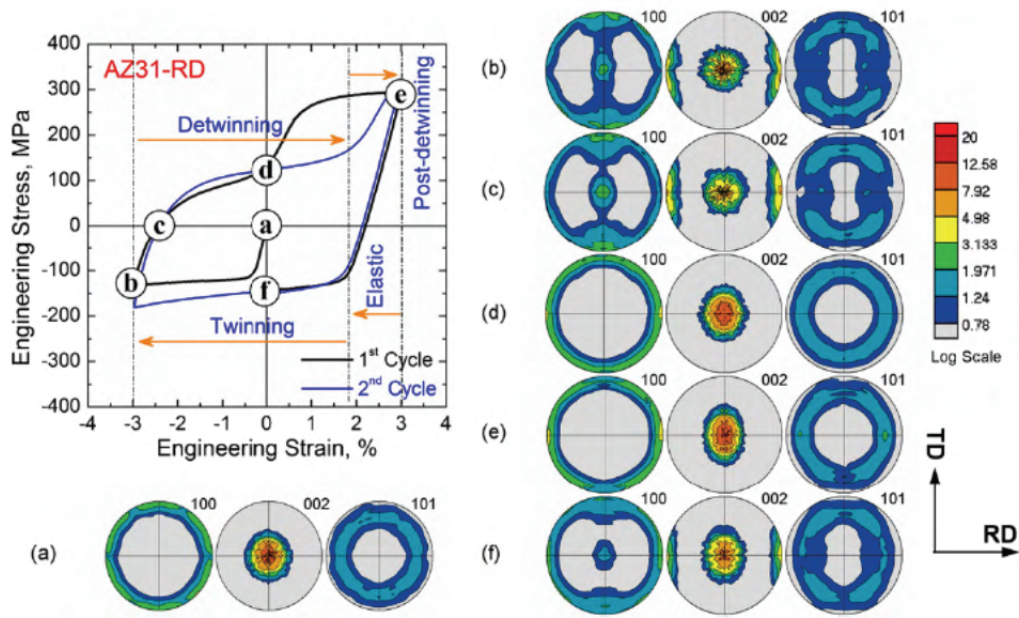


Figure 2-41 Measured crystallographic textures along the hysteresis loop during the first cycle deformation using ex-situ synchrotron diffraction: (a) strain-free, (b) -3.0% strain, (c) 0MPa stress, (d) 0% strain, (e) +3.0% strain, and (f) 0% strain. Pole densities are expressed in multiples of a random distribution (mrd). The loading axis (along RD) is horizontally right. [46]

### **2.6.2 Twin volume fraction measured by neutron diffraction**

Based on the pole figures, the axial distribution function, which is a cut through the pole figure at a line from the center to the perimeter can also be represented, in Figure 2-42 [50]. Considering the twinning makes the c-axes of the crystal rotates  $86.3^\circ$ , the significant axial distribution of  $\{0001\}$  is shown. During the compression along the extrusion bar of Mg alloy, the initial basal pole shifts from the rim to the center, where is the extrusion direction. Therefore, the peak intensity of axial distribution of basal pole shows at  $90^\circ$  at initial and gradually replaced by the peak shown at  $0^\circ$  with the increase in the strain. Therefore, the volume fraction of the extension twin can be obtained through integration of the area under the axial distribution. In this case, the integration area from  $0-45^\circ$  is the volume twinning, while the area from  $45-90^\circ$  can represent the volume of the parent.

The intensity of pole transferring from the  $Q_\perp$  to the  $Q_\parallel$  detector, as shown schematically in Figure 2-43 [59] is another way to obtained the volume fraction of extension twin during the compression along the extrusion direction in this case due to the geometric relationship. The prismatic pole diffraction intensity transfers from the  $Q_\parallel$  detector to  $Q_\perp$ . The transfer of intensity between the two detector banks is a direct measure of the twinned volume fraction and allows us to monitor the development of the twinned microstructure as well.



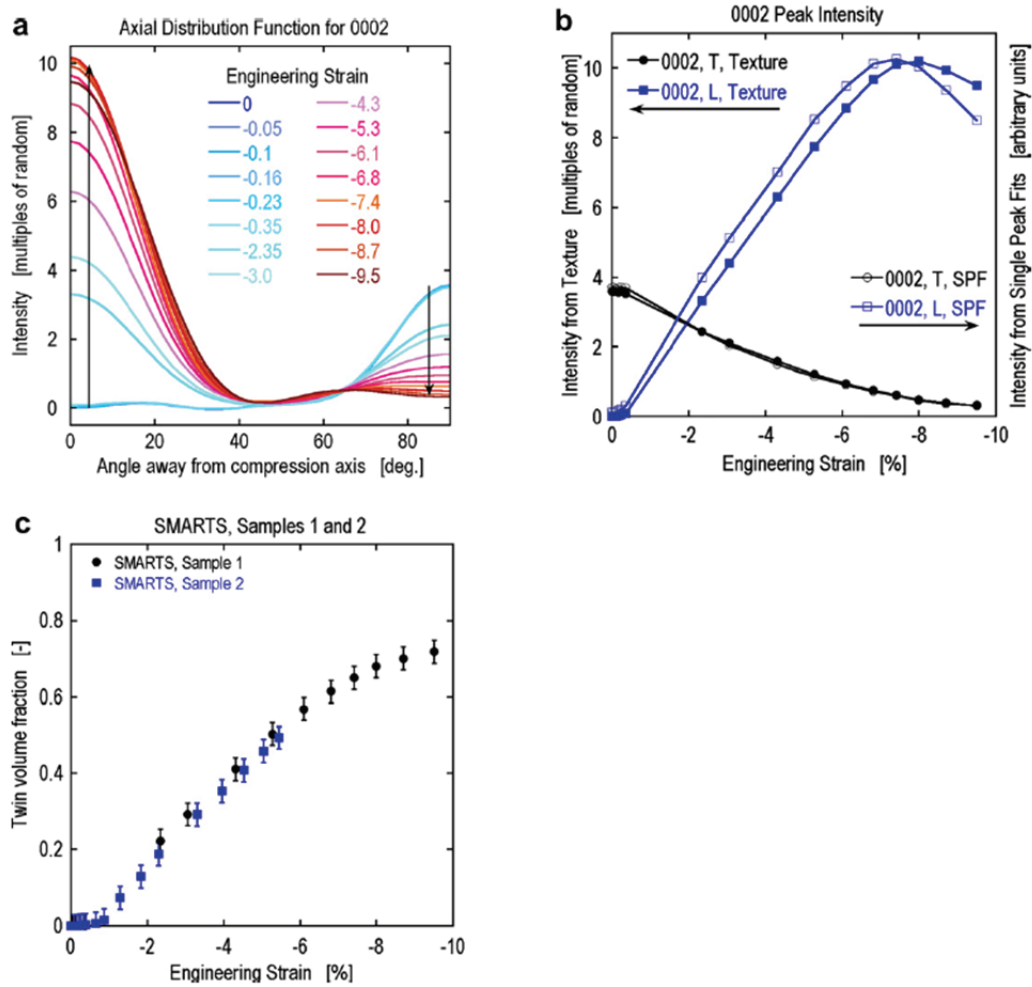


Figure 2-42 Measured axial distributions determined assuming fiber symmetry as a function of engineering strain. Notice the large jump in strain between  $-0.35\%$  and  $-2.35\%$ . (b) 0002 peak intensity as a function of engineering strain; extracted from the 0002 poles shown in (a) and from single peak fits. (c) Twin volume fraction obtained by integrating the area under the curves in (a).[50]

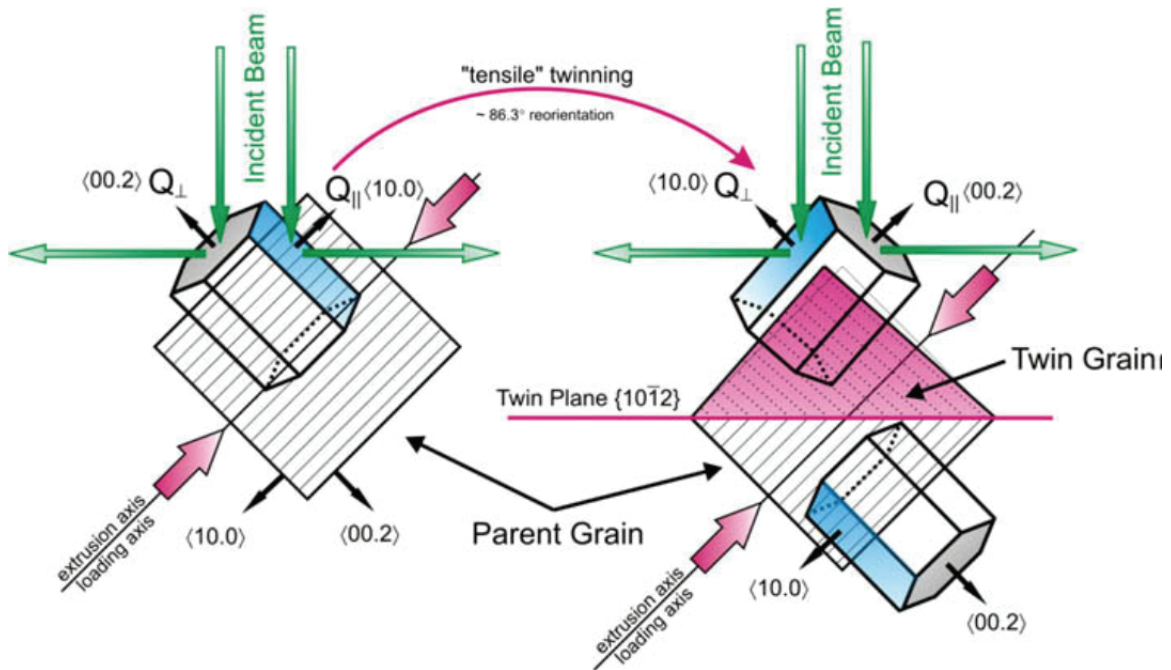


Figure 2-43 Schematic drawing of the parent  $\{10.0\}_{\parallel}$  grain (left) with associated twin  $\{00.2\}_{\parallel}$  grain (right) in respect to the loading/extrusion axis and the time-of-flight diffraction geometry used in the experiment. The figure schematically shows the reorientation of the parent  $\{10.0\}_{\parallel}$  lattice during tensile deformation twinning and the principle of its investigation using the in-situ neutron diffraction technique. The right figure also indicates the geometrical shortening of the twinning grain (i.e.  $\{10.0\}_{\parallel}$  parent grain) along the loading axis.[59]

### 2.6.3 The lattice strain measurement by neutron diffraction

The in-situ neutron diffraction technique measures the lattice spacing  $d_{hkl}$  of the planes (hkl) in a subset of grains that have the (hkl) plane normal parallel to the diffraction direction. The lattice strain is determined by:

$$\langle \varepsilon_{hkl} \rangle = \frac{d_{hkl} - d_{hkl,0}}{d_{hkl,0}} \quad 2-44$$

, where  $d_{hkl,0}$  is the initial (stress – free) plane spacing in the single crystals. Actually, the lattice strains are a measurement of the average elastic normal strain in the direction of the scattering vector in grains whose (hkl) lattice plane normal is parallel to the scattering vector. Hence, it does not represent the state within a single grain or the bulk, but an average over a subset of grains which fulfill the Bragg scattering condition for a given reflection. It worth to note that the accuracy of all lattice strain measurements highly depends on the accuracy of the  $d_{hkl,0}$  values. However, some of the diffraction peaks are not initially present in both banks as mentioned above and an alternative method of determining the initial lattice spacing,  $d_{hkl,0}$ , is needed for those planes. In this case, the ratio between a given (hkl) peak positions in bank 1 and bank 2 was determined separately for all the peaks present. For an unstressed material that is thermo-elastically isotropic and has not been subjected to prior plastic deformation, this ratio should be the same for all (hkl) peaks. Considering the low thermo-elastic anisotropy of magnesium alloys, the error is acceptable for the lattice measurement.

Wang [151] studied the lattice strain evolution in extruded magnesium alloy AZ31 under uniaxial tension through EVPSC. The results are compared against in-situ

neutron diffraction measurements done on the same alloy, Figure 2-44 [151]. Based on the lattice strain measurement, the input constitutive parameters of EVPSC model is modified. Moreover, for the first time, the effects of stress relaxation and strain creep on lattice strain measurements in respectively displacement controlled and load controlled in-situ tests are numerically assessed. It is found that the stress relaxation has a significant effect on the lattice strain measurements.

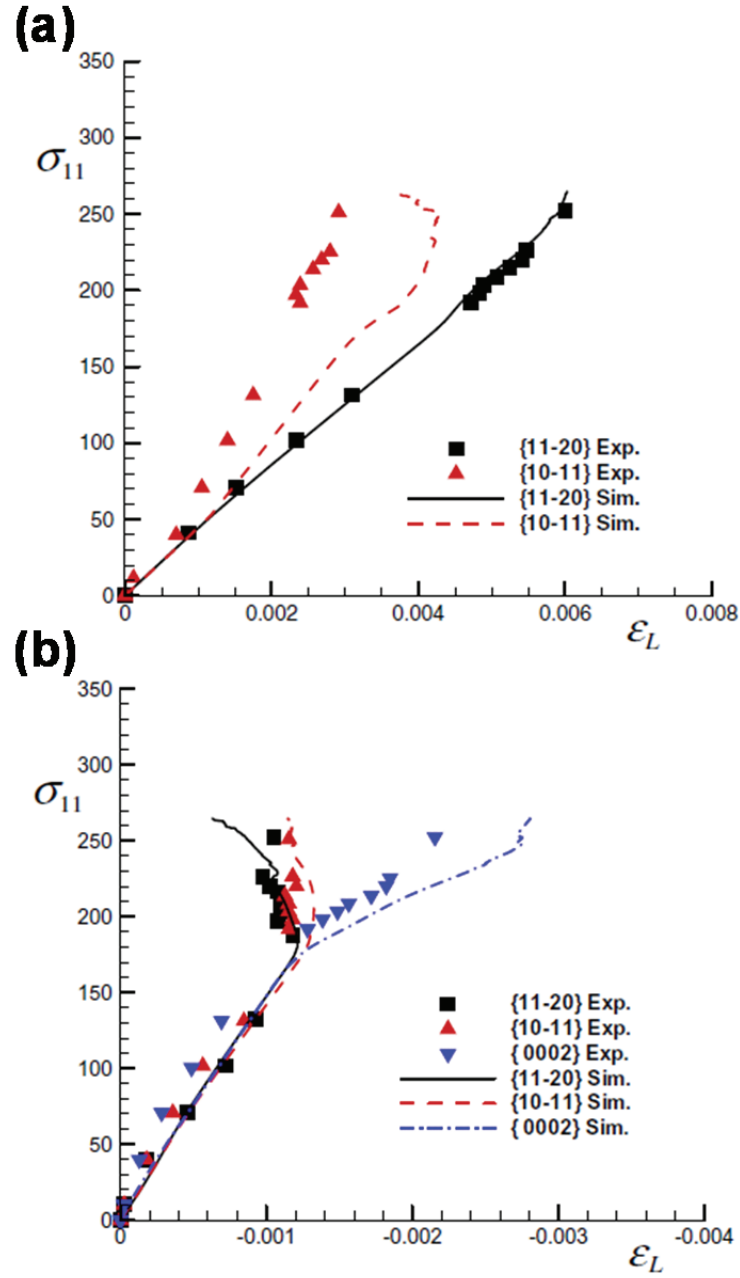


Figure 2-44 Simulated and experimental lattice strains under uniaxial tension (a) along the axial direction, and (b) along the transverse direction [151]

## **Chapter 3**

### **Influence of Texture on Hall-Petch Relationships in a Mg Alloy**

This chapter is written based on a paper published by Yi Wang, and Hahn Choo, “Influence of texture on Hall–Petch relationships in an Mg alloy”, *Acta Mater.* 2014; 81:83. As the first author, my primary contributions include (1) proposing the study and identifying its importance, (2) design and conducting most of the experiments, (3) data analysis and interpretation, and (4) writing the paper.

**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,  $\sigma_o$ , 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.

### 3.1 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 [152, 153]. During thermo-mechanical processes, the grain size can be refined through dynamic recrystallization (DRX) as a result of severe plastic deformation at elevated temperatures [154, 155]. For example, FSP, a solid-state joining process, can significantly refine the grain size in the stir zone [156, 157], depending on the processing parameters [2, 158]. Moreover, the crystallographic texture is often significantly altered during thermo-mechanical processes [88, 91]. According to Yu [159], 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:  $\{0001\} < 11\bar{2}0 >$  basal slip,  $\{10\bar{1}0\} < 11\bar{2}0 >$  prismatic slip,  $\{11\bar{2}2\} < 11\bar{2}\bar{3} >$  pyramidal slip, and  $\{10\bar{1}2\} <$

$10\bar{1}1 >$  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 [66, 67, 71]. For example, Agnew [160] and Jain [89] 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 [93] 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:

$$\sigma_y = \sigma_o + k_\sigma D^{-1/2} \quad 3-1$$

where  $\sigma_y$  is the yield strength,  $\sigma_o$  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 [161]. On the other hand, the effect of grain size on twinning is still not very well understood. Meyer [122] 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 [129] 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 [162], 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 [163]. Thus, different deformation systems should result in different H-P relations. Indeed, a current review of literature [45, 61, 95, 96, 99-117, 119-121, 164] shows that large discrepancies in the values of H-P parameters are evident as summarized in Table 2-6. The H-P parameters vary widely even for the same alloy AZ31B: the grain size sensitivity  $k_\sigma$  (MPa·mm<sup>1/2</sup>) varies from 5.0 to 12 and the friction stress  $\sigma_o$  (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 [119]. The H-P parameters are particularly sensitive to the loading path, which is illustrated by comparing tension [108] and compression [113] 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 chapter, 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,  $\sigma_o$ , 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.

## **3.2 Experimental**

**3.2.1 Material, heat treatment, and microstructure** The material used in this study is a commercial AZ31B Mg alloy, which was hot rolled with O-temper annealing condition. 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 chapter, 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 [165] 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.

### **3.2.2 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  $\omega$  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 [166] for texture presentation in terms of pole figures. Further details of the texture measurement can be found in [167].

### **3.2.3 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 Figure 3-1. The angle,  $\theta$ , 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  $10^{-3}\text{s}^{-1}$ . 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  $\pm 0.5\%$ .

### **3.3 Results**

#### **3.3.1 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 26 $\mu\text{m}$ , Figure 3-2a. Figure 3-2b 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.

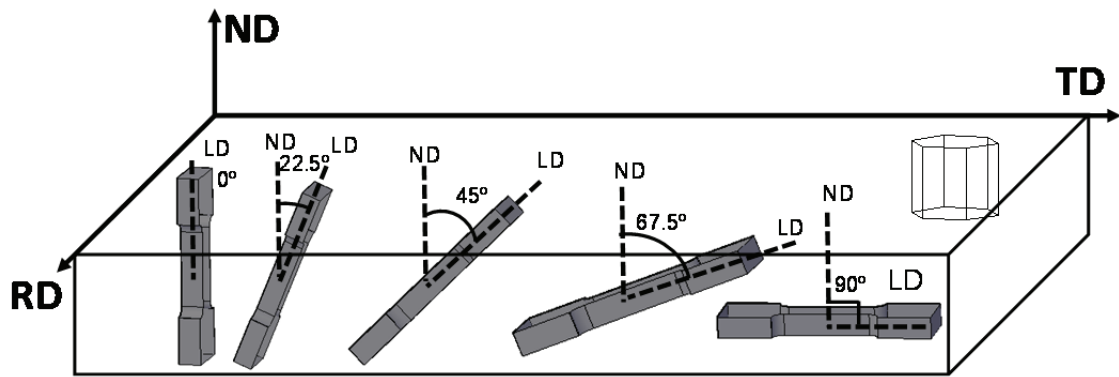


Figure 3-1 A schematic illustrating the preparation of tensile specimens. A total of five different orientations were prepared with the angle,  $\theta$ , between the LD and ND changing from  $0^\circ$ ,  $22.5^\circ$ ,  $45^\circ$ ,  $67.5^\circ$ , to  $90^\circ$ . ND, TD, RD, and LD refer to normal, transverse, rolling, and tensile loading direction, respectively.

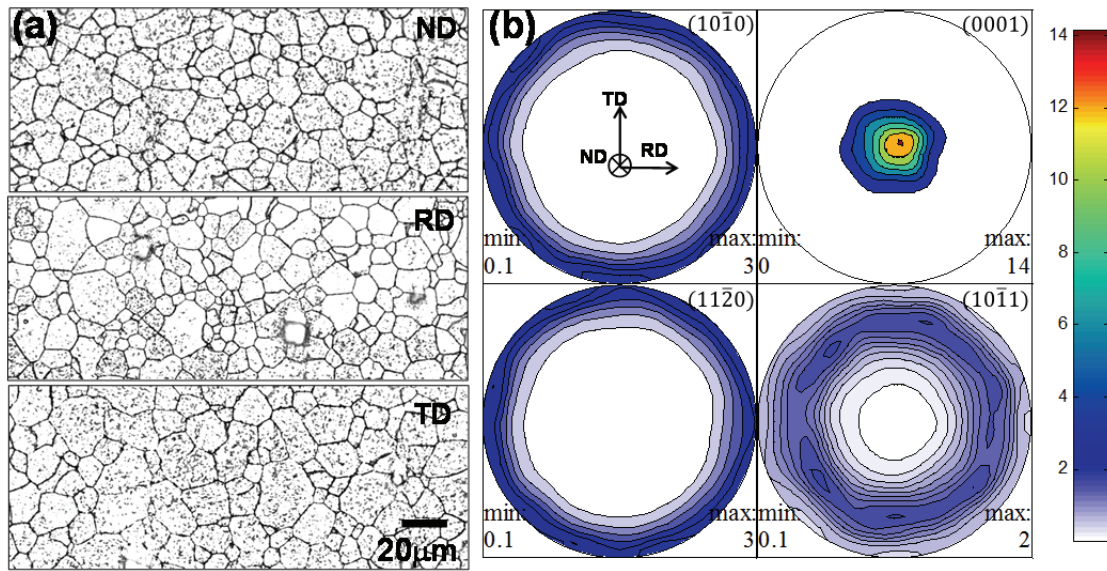


Figure 3-2 Grain size and texture of the as-received AZ31B Mg rolled plate: (a) optical micrographs from ND, RD, and TD surfaces. (b)  $\{10\bar{1}0\}$ ,  $\{0001\}$ ,  $\{11\bar{2}0\}$ , and  $\{10\bar{1}1\}$  pole figures (PFs) measured using S-XRD.

### 3.3.2 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 OM's of the ND cross-sections for a few selected specimens are presented in Figure 3-3. 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 3-1. 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:

$$K = A \cdot \exp\left(-\frac{Q}{RT}\right) \quad 3-2$$

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

$$[168]: K = \frac{D^N - D_o^N}{t} \quad 3-3$$

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



Table 3-1 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 ( $\mu\text{m}$ ) |           | $Q$ (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                                                           |

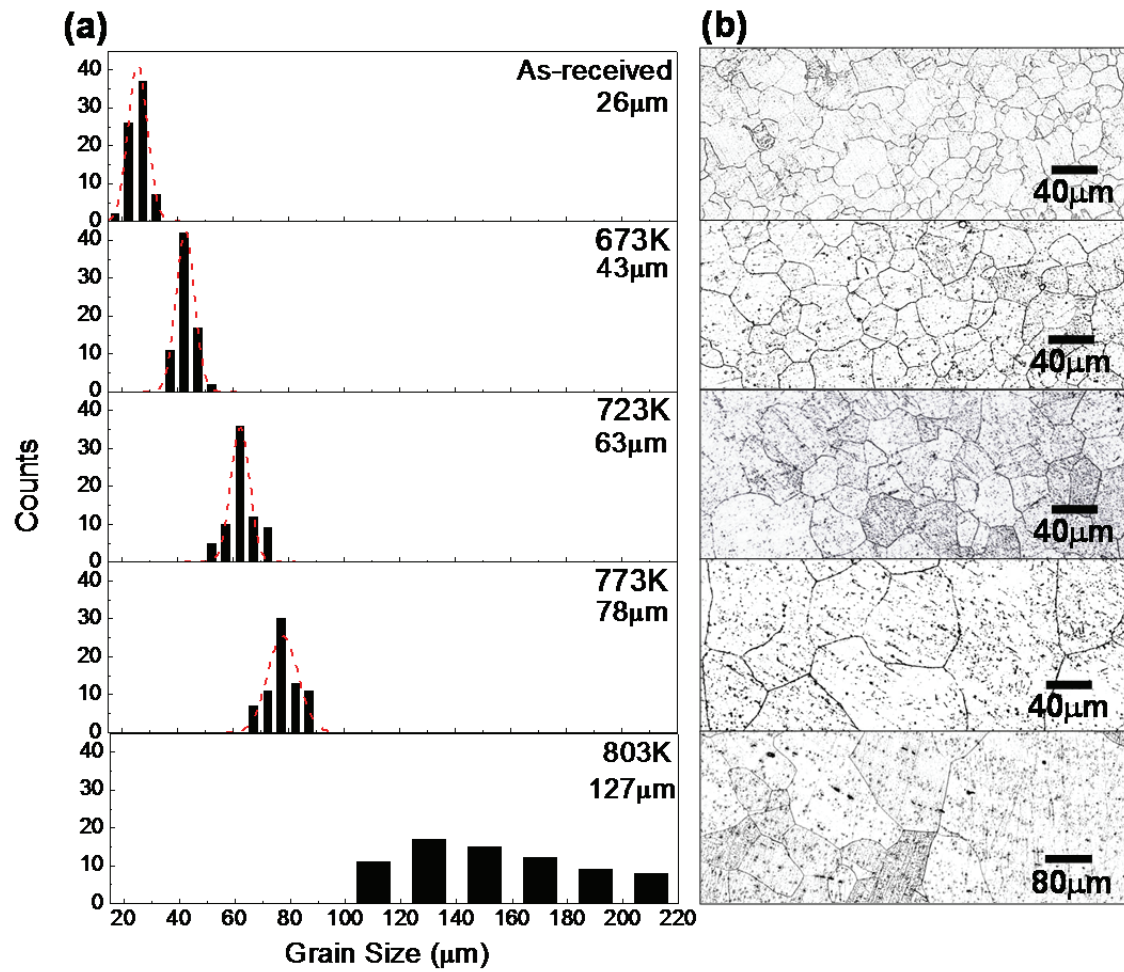


Figure 3-3 Grain size distributions and optical micrographs of AZ31B Mg plate after annealing at different temperatures from 673K to 803K for 96 hours: (a) grain size distributions with the Gaussian fitting. Also shown are the annealing temperature and mean grain size. (b) optical micrographs from the ND surface.

$$\ln \left( \frac{D^N - D_o^N}{t} \right) = \ln A - Q \cdot \frac{1}{RT} \quad 3-4$$

The measured grain growth kinetics of AZ31B Mg alloy is shown in Figure 3-4. For a pure metal, grain growth component  $N$  is 2 [169]; but it is usually higher than 2 for an alloy. It was reported that  $N$  ranges from 2 to 8 for Mg alloys [114]. 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 (Figure 3-4 and Table 3-1), which is comparable to the activation energy for the boundary diffusion (92 kJ/mol) for pure Mg [170]. Thus, the boundary diffusion could be the dominant mechanism for the grain growth below 773K. Above 773K, the activation energy increases to 256 kJ/mol, which indicates a secondary recrystallization process [171]. The secondary recrystallization is often related to a presence of second-phase particles, texture, or surface effects [85]. In the current hot-rolled Mg plate, there are a large number of low-angle grain boundaries with the strong plane texture. 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, the integrated intensities of {0002} reflections were compared between ND and RD, Table 3-1. The intensity ratio is reasonably constant up to 773K.

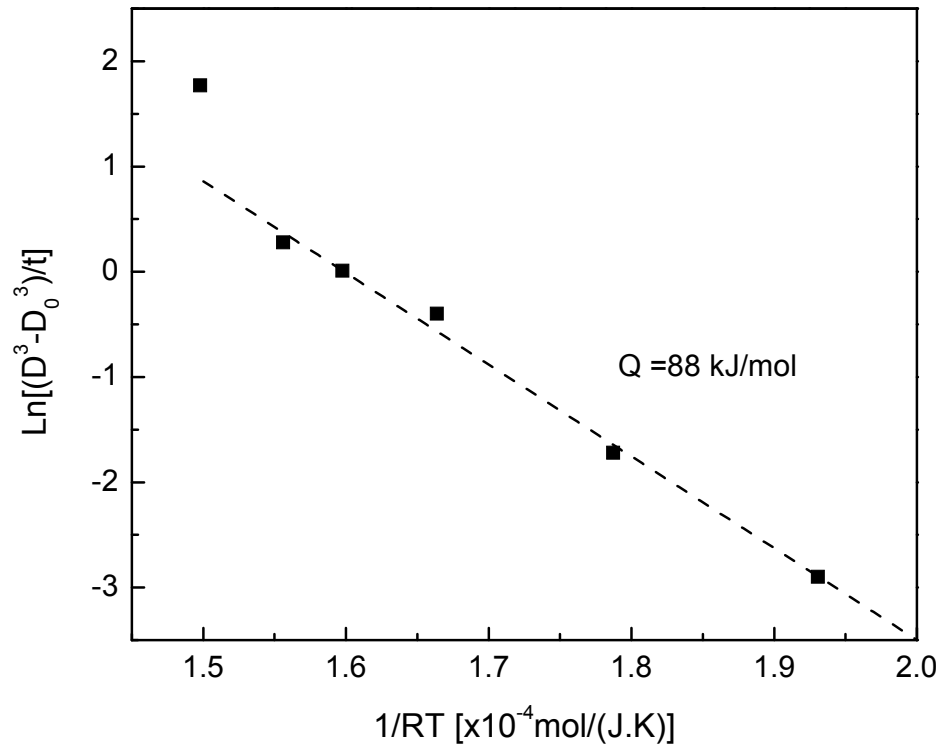


Figure 3-4 Grain growth kinetics of AZ31B Mg alloy during isochronal annealing of 96 hours at temperatures ranging from 623K to 803K. The initial grain size,  $D_0$ , is 26 $\mu\text{m}$ .

However, the texture is significantly altered at 803K, consistent with the abnormal grain size distribution and changes in the 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, Figure 3-5. 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.

### **3.3.3 Tensile behavior as a function of $\theta$**

Figure 3-6 shows tensile stress-strain curves for the as-received specimens ( $D \approx 26\mu\text{m}$ ) measured as a function of  $\theta$ , the angle between the tensile LD and ND of the Mg plate. It is clearly shown that the yielding (inset in Figure 3-6) and hardening behaviors change with the changes in the tensile orientation.

For the  $0^\circ$  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.

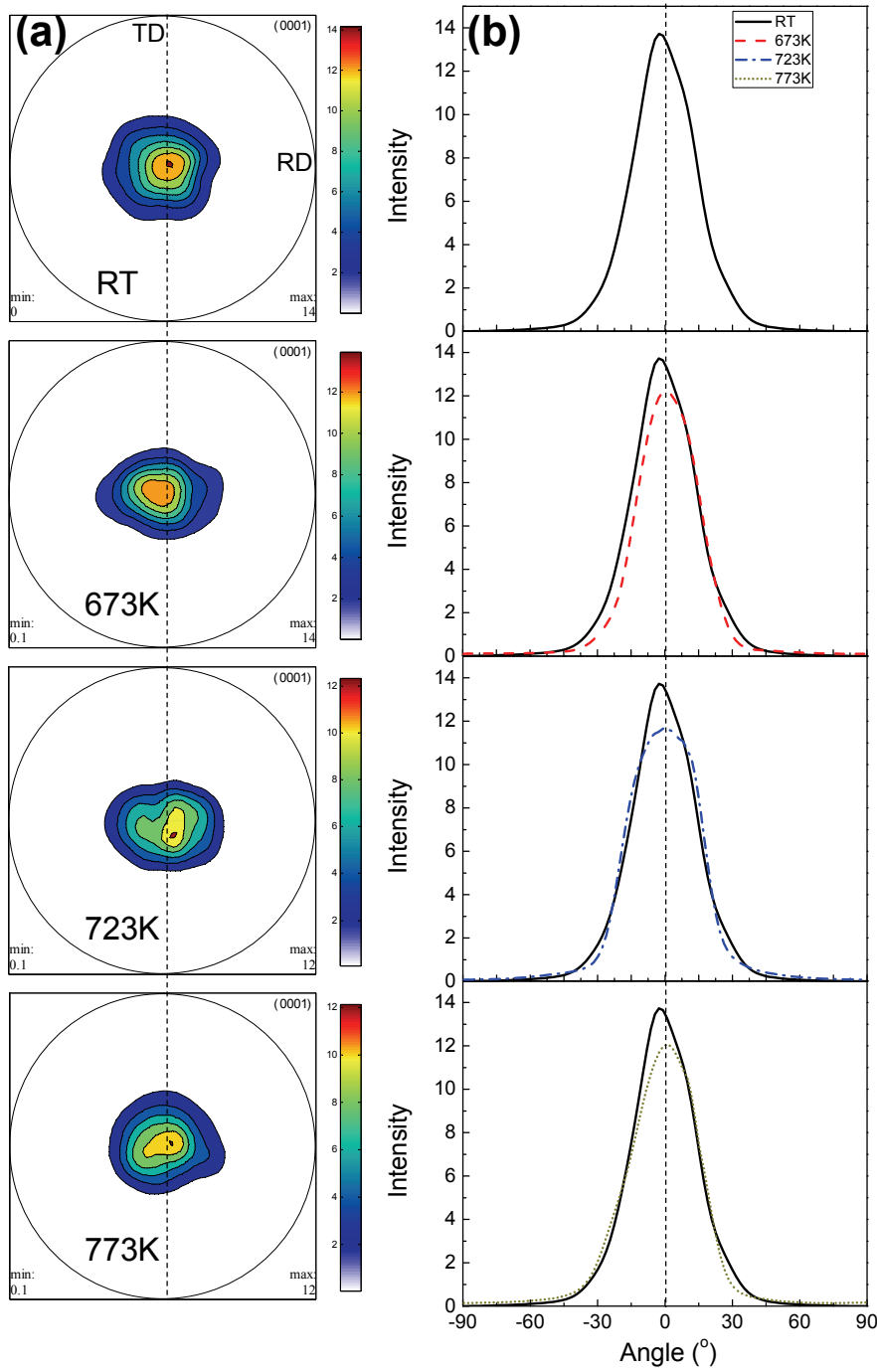


Figure 3-5 Textures measured after annealing at different temperatures from 673K to 773K compared to the as-received condition: (a) {0001} pole figures, and (b) axial distribution of {0001} intensity from ND to TD in the {0001} PFs.

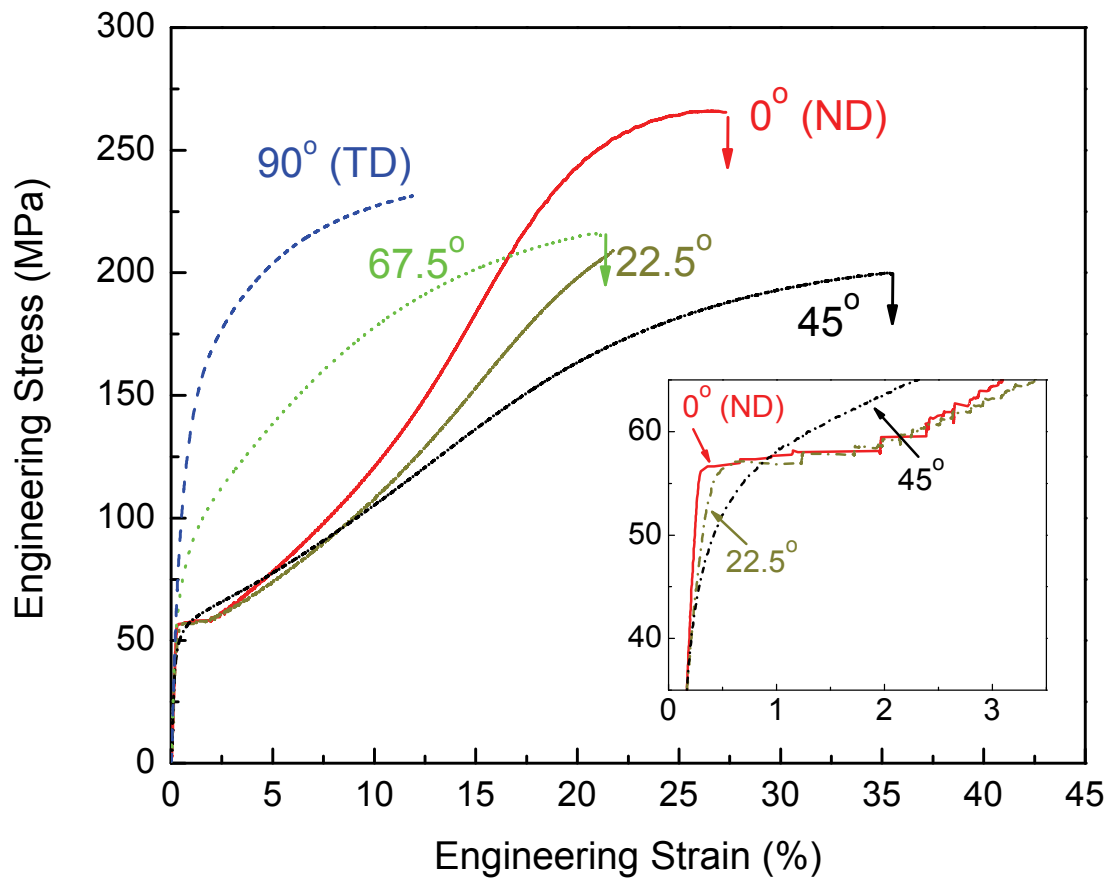


Figure 3-6 Tensile stress-strain curves of the as-received Mg alloy as a function of the angle,  $\theta$ , between the tensile loading direction (LD) and normal direction (ND) of the plate. Also shown in the inset are the yielding behaviors of  $0^\circ$ ,  $22.5^\circ$ , and  $45^\circ$  specimens.

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  $0^\circ$  case is a manifestation of an extension-twinning dominant plastic deformation, similar to the compression along TD or RD of a rolled sheet [38, 42] or compression along the extrusion direction of an extruded bar [33]. The appreciable work hardening with the increase in strain is caused primarily by the crystal reorientation as a result of the extension twin [33]. The stress-strain behavior of the  $22.5^\circ$  case is similar to that of  $0^\circ$ , in that the plateau stage occurs after the yielding, while the subsequent hardening rate is slightly lower than that of the  $0^\circ$  case.

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

For the  $67.5^\circ$  and  $90^\circ$  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  $67.5^\circ$  case is between those of  $45^\circ$  and  $90^\circ$  cases.

#### ***3.3.4 Hall-Petch relationship as a function of $\theta$***

Figure 3-7 shows the tensile stress-strain behaviors of specimens with four different grain sizes for each loading orientation,  $\theta$ . Also shown in the insets are magnified views of the yield behavior for different grain sizes.



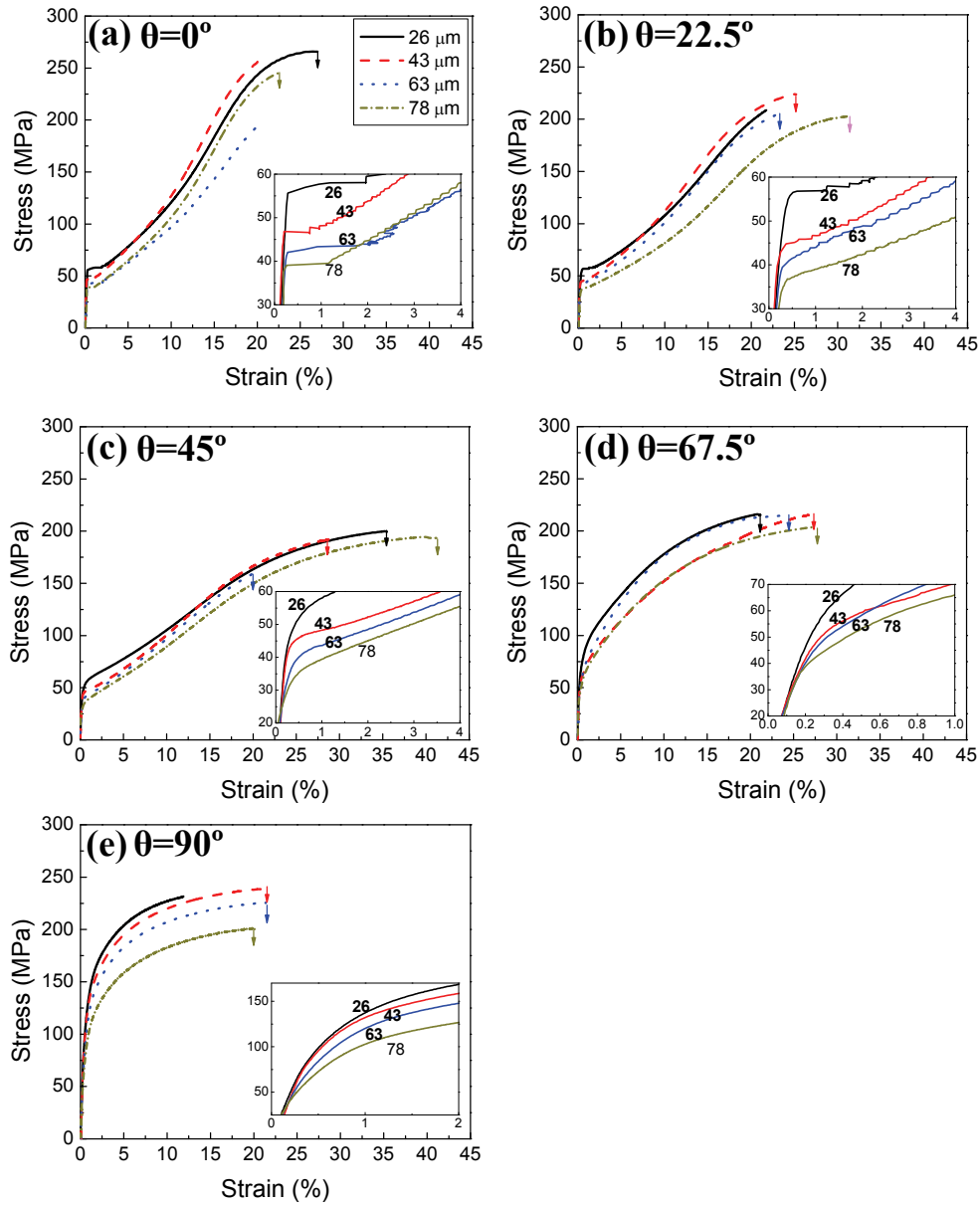


Figure 3-7 Tensile stress-strain behaviors of the specimens with four different grain sizes ranging from 26 $\mu\text{m}$  to 78 $\mu\text{m}$  as a function of the angle,  $\theta$ , between the LD and ND: (a)  $\theta=0^\circ$  loading case; (b)  $22.5^\circ$ ; (c)  $45^\circ$ ; (d)  $67.5^\circ$ ; and (e)  $90^\circ$ . Also shown in the insets are the yield behaviors of specimens with different grain sizes.

The characteristic yielding and hardening behaviors for each  $\theta$  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 discussed in the following chapters 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 Figure 3-8. 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  $\theta$ . In Table 3-2, different intercepts ( $\sigma_o$ ) and slopes ( $k_\sigma$ ) obtained for each  $\theta$  are summarized. The measured  $\sigma_o$  ranges from 10.5 to 41.1 (MPa) and  $k_\sigma$  ranges from 5.0 to 13.0 (MPa·mm<sup>1/2</sup>) for different loading orientations. They are in good agreement with the wide ranges reported in Table 2-6. It is shown that the  $k_\sigma$  is lowest for the tension along 45°; while the 90° case exhibits the highest  $k_\sigma$  and  $\sigma_o$ , implying the strongest grain size sensitivity.

Table 3-2 Hall-Petch parameters ( $\sigma_o$  and  $k_\sigma$ ) measured as a function of the angle,  $\theta$ , between the LD and ND.

| Angle, $\theta$ (Deg.) | Hall-Petch Parameters |                                             | $R^2$ |
|------------------------|-----------------------|---------------------------------------------|-------|
|                        | $\sigma_o$ (MPa)      | $k_\sigma$ (MPa $\cdot$ mm <sup>1/2</sup> ) |       |
| 0                      | 12.2 ( $\pm$ 1.2)     | 7.2 ( $\pm$ 0.2)                            | 0.996 |
| 22.5                   | 10.5 ( $\pm$ 1.1)     | 7.3 ( $\pm$ 0.2)                            | 0.996 |
| 45                     | 18.2 ( $\pm$ 0.3)     | 5.0 ( $\pm$ 0.07)                           | 0.999 |
| 67.5                   | 26.5 ( $\pm$ 3.6)     | 7.0 ( $\pm$ 0.7)                            | 0.965 |
| 90                     | 41.1 ( $\pm$ 6.6)     | 13.0 ( $\pm$ 1.4)                           | 0.964 |

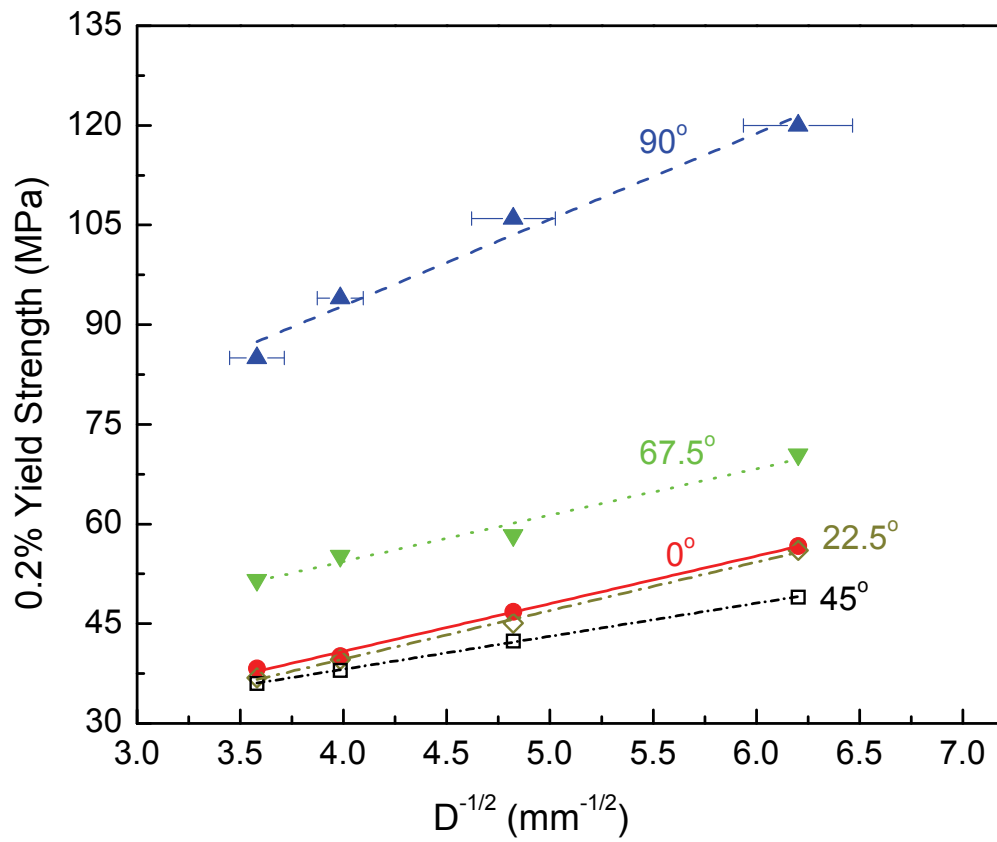


Figure 3-8 Hall-Petch relationships established as a function of the angle,  $\theta$ , between the LD and ND.

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.

### 3.4 Discussion

#### 3.4.1 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 [48].

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

$$\sigma_y = \tau_{CRSS}/m \quad 3-5$$

, where  $\tau_{CRSS}$  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  $\{0001\} < 10\bar{1}0 >$ , Schmid factors for the various deformation modes can be calculated for different LDs (see Table 3-3). In the “ideal” Schmid factor calculation, two points are worth noting.

Table 3-3 Ideal/measured Schmid factors,  $m$ , for basal slip, prismatic slip, pyramidal slip, and extension twin as a function of the tensile loading orientation,  $\theta$ , between the LD and ND. Ideal Schmid factor is calculated assuming a perfect  $\{0001\} < 10\bar{1}0 >$  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.

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

First, to simplify the calculation, it was assumed that the rolled Mg plate has the ideal  $\{0001\} \langle 10\bar{1}0 \rangle$  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 3-3 are the upper boundary values, and the calculated  $\sigma_y$  using such  $m$  values would provide lower boundaries. Also, the average “measured” Schmid factors for each deformation mode are obtained from the texture measured by S-XRD (Table 3-3). The measured values show slightly smoother changes as a function of  $\theta$  since the distribution of  $\{0002\}$  pole from the ND are taken into account.

For the  $0^\circ$  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  $\theta$ ,  $m$  of extension twin continuously decreases. When  $\theta$  is higher than  $47^\circ$ , the extension twin is impossible to be activated with  $c/a$  ratio close to or smaller than 1.633. For the  $45^\circ$  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  $\theta$  is  $90^\circ$ . Therefore, only prismatic slip is able to accommodate the plastic deformation, which is consistent with Jiang [48], Barnett [67], and Koike’s [51] 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  $90^\circ$  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  $\theta$  is consistent with Oppedal's [72] and Jain's [35] studies using elasto-plastic self-consistent (EPSC) and VPSC simulations of the rolled Mg plate.

### 3.4.2 $\sigma_o$ as a function of $\theta$

In the H-P relationship,  $\sigma_o$  is the friction stress for dislocation movement, at which material yields if the grain-boundary effects are not considered. The significant variations in  $\sigma_o$  with the changes in  $\theta$  are clearly shown in Table 3-2. According to Armstrong [118], the  $\sigma_o$  is related to  $\tau_{CRSS}^\beta$  (CRSS of a given deformation mode  $\beta$ ) and orientation factor  $M$ :

$$\sigma_o = M \cdot \tau_{CRSS}^\beta \quad 3-6$$

In this section, the friction stress,  $\sigma_o$ , will be calculated as a function of  $\theta$  using Equation 3-6 based on the measured average Schmid factors (Table 3-3) and representative CRSSs for each deformation mode from the literature [67, 74] (Table 3-4). Then, the calculated  $\sigma_o$  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 [62] and various plastic deformation modeling methods such as EPSC, VPSC, or Sachs polycrystal models [67, 74, 137].



Table 3-4 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  $\tau_{CRSS}^{\beta}$ ), their ratios with respect to the basal slip, shear moduli, and Burgers vectors [67, 74, 172].

| 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     | -              |

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 [74, 117]. 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  $\{10\bar{1}2\}$  twinning, basal slip, and prismatic slip at  $0^\circ$ ,  $45^\circ$ , and  $90^\circ$ , respectively, are all close to 0.5 (Table 3-3), the differences in  $\sigma_o$  shown in Table 3-2 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  $\sigma_o$  (Table 3-4) show good agreements with the  $\sigma_o$  measured at  $\theta=0^\circ$ ,  $45^\circ$ , and  $90^\circ$ , respectively, with a similar ratio of:

$$\tau_{CRSS}^{Basal} : \tau_{CRSS}^{Prism} : \tau_{CRSS}^{Twin} = 1 : 2.0 : 0.7$$

The friction stress,  $\sigma_o$ , is calculated for different deformation modes for the five different orientations, and the results are presented in Table 3-5. The dominant deformation modes are revealed for each  $\theta$  by comparing the value of  $M \cdot \tau_{CRSS}^\beta$  for all possible modes. The minimum calculated  $\sigma_o$  in each loading orientation agrees well with the measured  $\sigma_o$  in that particular orientation.

Table 3-5 Calculated  $\sigma_o$  for different deformation modes for the five loading orientations,  $\theta$ , based on the measured Schmid factors (Table 3-3) and CRSSs from literature (Table 3-4). The minimum value in each orientation is shown in bold indicating the dominant plastic deformation mode at yielding.

| Angle, $\theta$<br>(Deg.) | Calculated $\sigma_o$ (MPa) |             |           |                   | Dominant Plastic<br>Deformation Mode at<br>Yielding |
|---------------------------|-----------------------------|-------------|-----------|-------------------|-----------------------------------------------------|
|                           | Basal                       | Prismatic   | Pyramidal | Extension<br>Twin |                                                     |
| 0                         | 61.4                        | 588.2       | 85.7      | <b>15.1</b>       | Twin                                                |
| 22.5                      | 30.7                        | 625.0       | 85.1      | <b>16.5</b>       | Twin (+Basal slip)                                  |
| 45                        | <b>21.4</b>                 | 175.4       | 131.1     | 26.6              | Basal slip (+Twin)                                  |
| 67.5                      | <b>26.7</b>                 | 72.5        | 99.0      | 93.3              | Basal slip                                          |
| 90                        | 128.2                       | <b>46.7</b> | 93.7      | -                 | Prismatic slip                                      |

For the  $0^\circ$  and  $22.5^\circ$  cases, the calculated  $\sigma_o$  for twinning is the minimum. The basal slip is the easiest one to be activated at  $45^\circ$  and  $67.5^\circ$ . 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  $\sigma_o$  is higher at  $67.5^\circ$  than at  $45^\circ$  because it is a slightly harder orientation with a relatively lower Schmid factor. At  $90^\circ$ , prismatic slip has the lowest  $\sigma_o$ .

Moreover, Figure 3-9 shows both the calculated (curves) and measured (symbol)  $\sigma_o$  as a function of  $\theta$ . The error bars for the measured  $\sigma_o$  comes from the standard deviation of the linear fitting of the H-P relations. The lower boundaries of the calculated lines across the  $\theta$  represent dominant deformation mechanisms at yielding. The calculation indicates that from  $\theta=0^\circ$  to  $39^\circ$ , extension twinning is dominant, while basal slip will contribute mostly at yielding between  $\theta=0^\circ$  and  $83^\circ$ . The prismatic slip is dominant from  $83^\circ$  to  $90^\circ$ . Henceforth,  $0^\circ$ ,  $67.5^\circ$ , and  $90^\circ$  cases will be designated as extension twin, basal slip, and prismatic slip cases, respectively.

### 3.4.3 $k_\sigma$ as a function of $\theta$

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  $3.5 \text{ MPa} \cdot \text{mm}^{1/2}$ ).

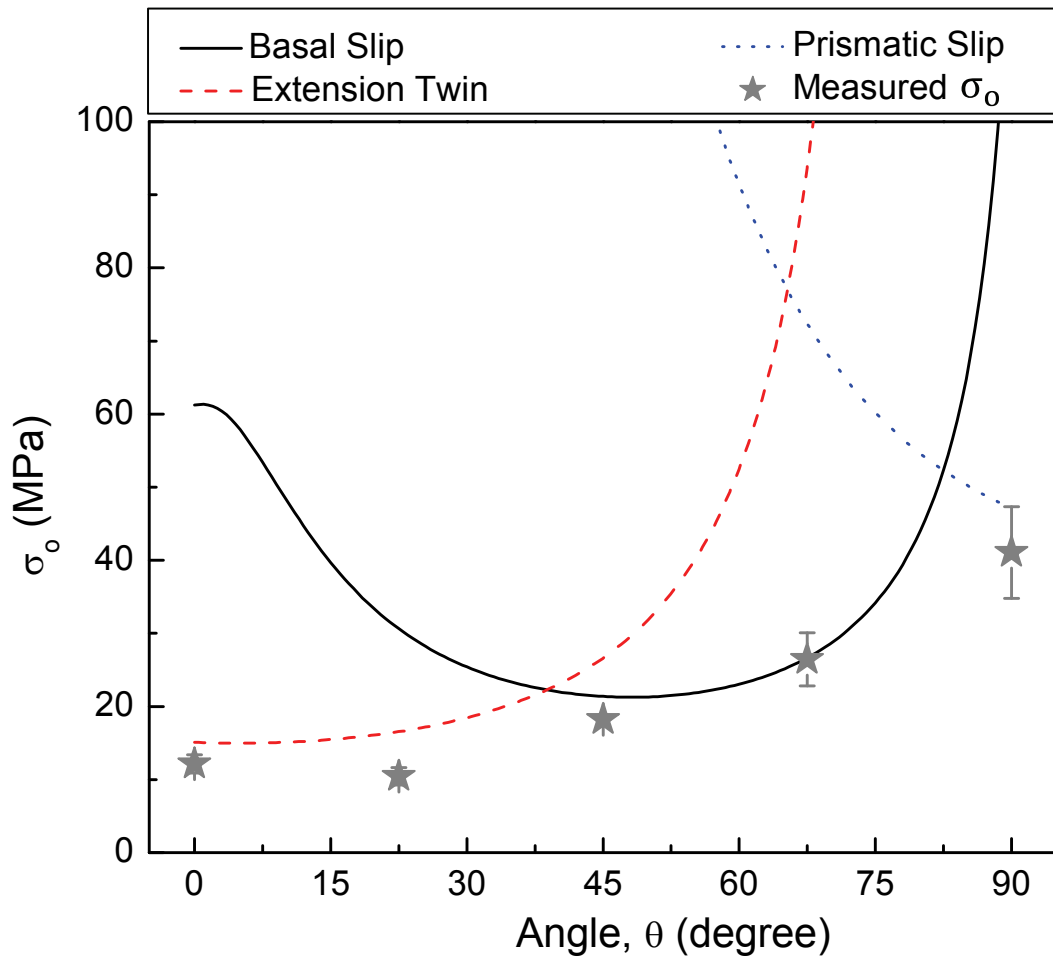


Figure 3-9 Calculated friction stress,  $\sigma_o$ , for different deformation modes as a function of the angle,  $\theta$ , between the LD and ND based on measured Schmid factors (Table 3-3) and the CRSSs from the literature (Table 3-4). Also shown in symbol are the measured  $\sigma_o$ .

The physical mechanisms of the H-P relationship discussed in the literature include: (1) dislocation pile-up theory [173], (2) GB source theory [174], (3) slip distance theory [175], and (4) geometrically-necessary dislocation theory [176]. One of the most commonly cited mechanisms for the grain-boundary effect is the dislocation pile-up theory proposed by Hall [177].

For Mg alloys, dislocation pile-ups near the grain boundaries have been investigated using transmission electron microscopy (TEM) [178, 179], 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 [161], the effective stress,  $\tau_e$ , acting on the moving dislocation is:

$$\tau_e = \tau - \tau_o \quad 3-7$$

where  $\tau$  is the applied shear stress and  $\tau_o$  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:

$$\tau_p = n\tau_e \quad 3-8$$

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

$$T_l = \frac{\tau_e b L_p}{n} \quad 3-9$$

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 3-8 can be rewritten as:

$$\tau_p = \frac{\tau_e^2 b L_p}{T_l} \quad 3-10$$

From Equations 3-7 and 3-10,

$$\tau_p = \frac{(\tau - \tau_o)^2 b L_p}{T_l} \quad 3-11$$

The dislocation line tension energy,  $T_l$ , can also be expressed as:  $T_l = \alpha G b^2$

$$3-12$$

where  $G$  is the shear modulus and  $\alpha$  is a constant. For screw dislocation,  $\alpha = 1/2\pi$ ; while for the edge dislocation,  $\alpha = 1/2\pi(1 - \nu)$ .  $\nu$  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 [180]. Also, for  $\langle c+a \rangle$  dislocation, screw dislocations glide on  $\{1\bar{1}01\}$ , while edge dislocations glide on  $\{11\bar{2}2\}$  planes according to the first principle simulation [181]. Accordingly,  $\alpha = 1/2\pi$  was used for the basal slip, while  $\alpha = 1/2\pi(1 - \nu)$  was used for the prismatic and pyramidal slips.

The dislocation can move across the GBs when the  $\tau_p$  is as high as the critical stress  $\tau_c$ . The applied shear stress  $\tau$  can be replaced by resolved shear stress,  $\tau_R$ , for a given grain size. Then, from Equations 3-11 and 3-12, the resolved shear stress,  $\tau_R$ , can be written as:

$$\tau_R = \tau_o + (\alpha \tau_c G b)^{1/2} \cdot D^{-1/2} \quad 3-13$$

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

$$k_\tau = (\alpha \tau_c G b)^{1/2} \quad 3-14$$

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,  $\tau_c$ . Finally, the H-P slope for yield strength,  $k_\sigma$ , can be written as:

$$k_\sigma = M(\alpha \tau_c G b)^{1/2} \quad 3-15$$

Using the CRSSs (as  $\tau_c$ ), shear moduli [172], Burgers vectors, and the inverse of the measured Schmid factors for basal and prismatic slip (Table 3-3 and Table 3-4),  $k_\sigma$  is calculated as a function of the loading orientation Figure 3-10. 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  $\theta$  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  $\theta$  are reflected well in the wide range of the H-P parameters reported in the literature (Table 2-6) for Mg alloys subjected to different thermo-mechanical processes and test conditions. For instance, Wang [108] reported that  $k_\sigma$  (MPa·mm<sup>1/2</sup>) 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.



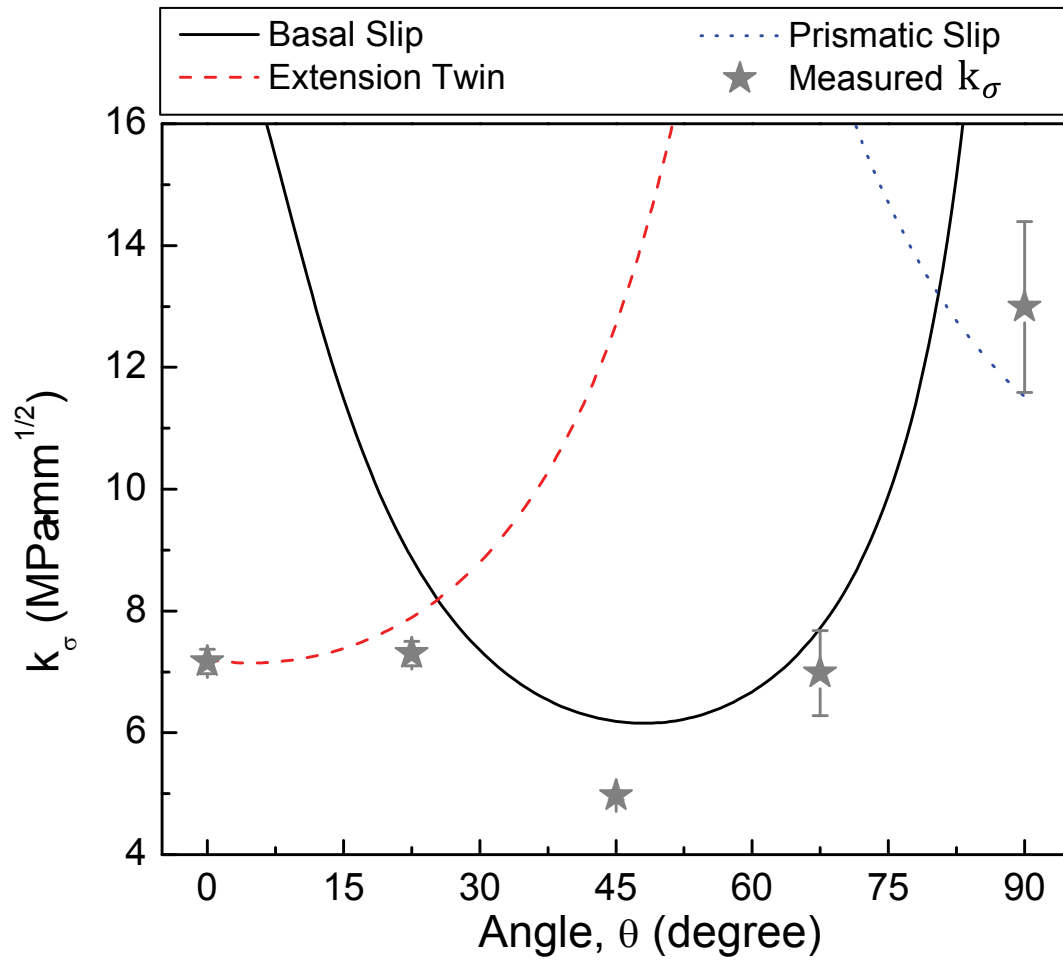


Figure 3-10 Calculated grain size sensitivity,  $k_\sigma$ , for different deformation modes as a function of the angle,  $\theta$ , between the LD and ND. Also shown in symbol are the measured  $k_\sigma$ .

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.

#### 3.4.4 Yield strengths as a function of grain size and loading orientation

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

$$\mathbf{a} = (\cos\theta, 0, \sin\theta) \quad 3-16$$

In the Cartesian coordinate system, slip plane normal  $\mathbf{b}$  and the slip direction  $\mathbf{c}$  for a given deformation mode can be expressed as:

$$\mathbf{b} = \begin{pmatrix} 1 & 0 & 0 \\ 1/\sqrt{3} & 2/\sqrt{3} & 0 \\ 0 & 0 & 1/1.624 \end{pmatrix} \cdot \mathbf{P}^\beta \quad 3-17$$

$$\mathbf{c} = \begin{pmatrix} 1 & -0.5 & 0 \\ 0 & \sqrt{3}/2 & 0 \\ 0 & 0 & 1.624 \end{pmatrix} \cdot \mathbf{D}^\beta \quad 3-18$$

where  $\mathbf{D}^\beta$  is the slip direction,  $\mathbf{P}^\beta$  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:

$$M = \frac{|\mathbf{a}| \cdot |\mathbf{b}|}{\mathbf{a} \cdot \mathbf{b}} \cdot \frac{|\mathbf{a}| \cdot |\mathbf{c}|}{\mathbf{a} \cdot \mathbf{c}} \quad 3-19$$

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

$$\sigma_y = M \left\{ \tau_o^\beta + \left[ \alpha G^\beta \tau_o^\beta b^\beta \right]^{1/2} \cdot D^{-1/2} \right\} \quad \text{Slip} \quad 3-20$$

$$\sigma_y = M \left( \tau_o^{twin} + \frac{M}{M(0)} \cdot k_\tau^{twin} \cdot D^{-1/2} \right) \quad \text{Extension twin} \quad 3-21$$

The tensile yield strength contours as a function of the grain size and loading orientation,  $\theta$ , with respect to the normal direction (i.e.,  $c$ -axis) of the hot-rolled Mg plate are presented in Figure 3-11 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.

#### 3.4.5 *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, Figure 3-12. The intercepts of the linear regression are  $\tau_o^\beta$  and the slopes represent the grain size sensitivity,  $k_\tau^\beta$ , for each deformation mode as summarized in Table 3-6. Moreover, the calculated  $k_\tau^\beta$  for basal, prismatic, and pyramidal slip according to the Equation 3-14 are also shown in Table 3-6.

Table 3-6 Hall-Petch parameters of various deformation modes for AZ31B Mg alloy.

| Def. Mode         | $\tau_o$ (MPa) | $k_{\tau}$ (MPa $\cdot$ mm <sup>1/2</sup> ) | Calculated $k_{\tau}$ (MPa $\cdot$ mm <sup>1/2</sup> ) |
|-------------------|----------------|---------------------------------------------|--------------------------------------------------------|
| Basal Slip        | 9.9            | 2.61                                        | 2.89                                                   |
| Prismatic Slip    | 17.6           | 5.54                                        | 4.88                                                   |
| Pyramidal<br>Slip | -              | -                                           | 9.86                                                   |
| Ex. Twin          | 5.7            | 3.48                                        | -                                                      |

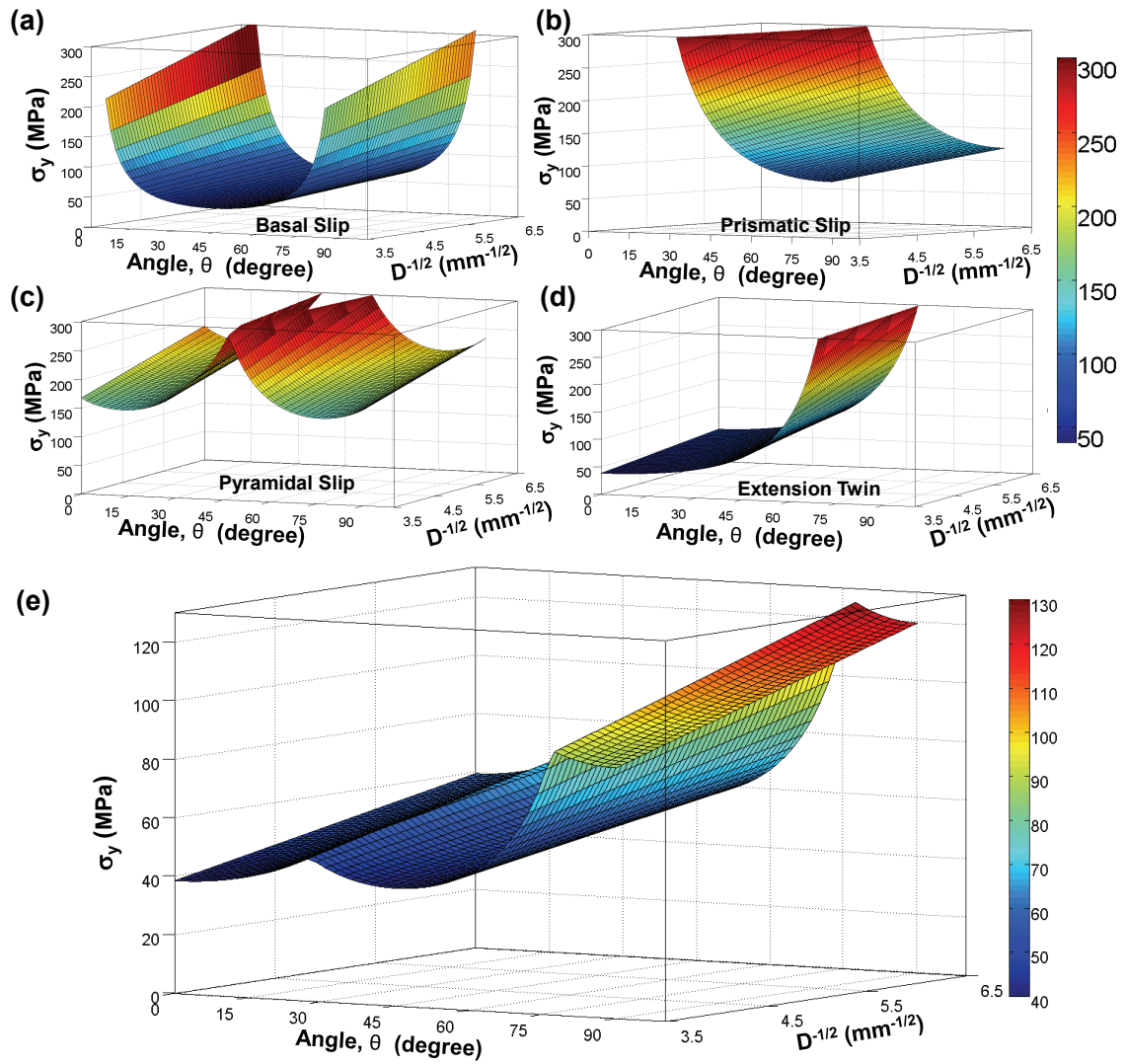


Figure 3-11 Yield strength contours of a hot-rolled AZ31B Mg plate as a function of the grain size and tensile loading orientation,  $\theta$ , with respect to the normal direction (ND) of the plate (i.e.,  $c$ -axis of the crystals). (a) Basal slip only, (b) prismatic slip only, (c) pyramidal slip only, (d) extension twinning only, and (e) yield surface when all deformation modes are considered.

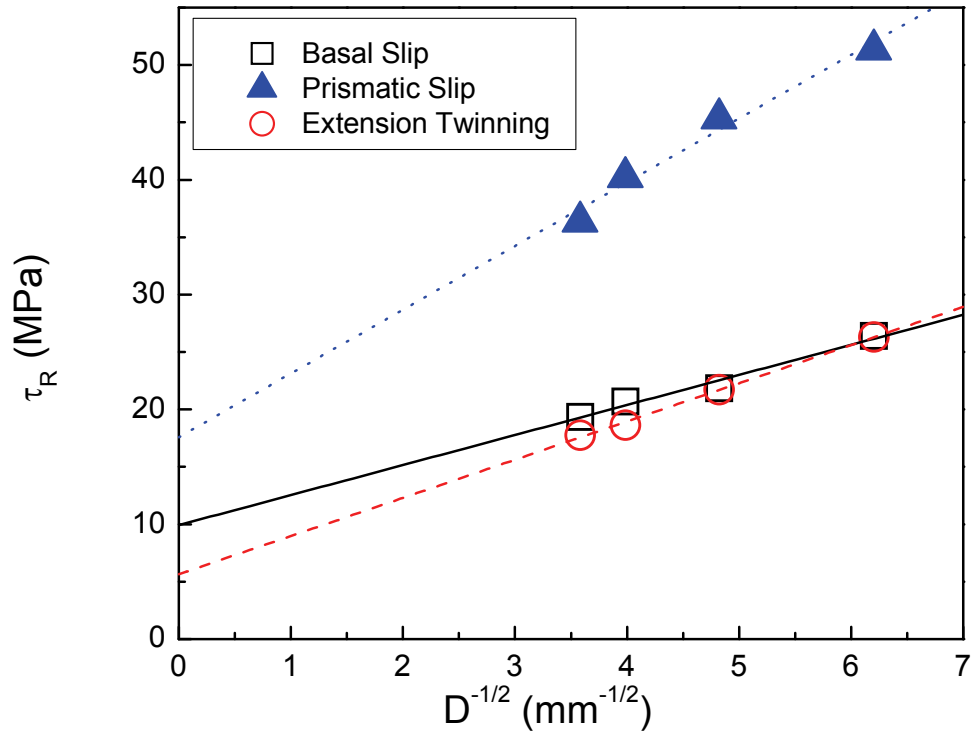


Figure 3-12 Hall-Petch relationships for basal slip, prismatic slip, and extension twinning for AZ31B Mg alloy in terms of the shear stress calculated from the yield strength and measured Schmid factor. The  $y$ -intercept  $\tau_o^\beta$  and the slope  $k_\tau^\beta$  are summarized in Table 3-6.

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. The VPSC modeling that incorporates H-P relationships will be discussed in Chapter 5.

Table 3-6 shows that the grain size sensitivity of pyramidal slip is the highest ( $9.86 \text{ MPa}\cdot\text{mm}^{1/2}$ ) 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 [51]. 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  $a/3 \langle 11\bar{2}0 \rangle$ . The shear moduli of the basal plane and prismatic plane are only slightly different because of the asymmetry of the HCP crystal [172]. Therefore,

differences in the grain size sensitivities for these two slip systems mainly come from the differences in  $\tau_c$ .

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 [129], 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].

### 3.5 Conclusions

The influence of texture on the Hall-Petch relationship is investigated using a commercial hot-rolled AZ31B Mg alloy plate with a strong  $\{0001\} < 10\bar{1}0 >$  plane texture. The texture variations are facilitated by changing the angle,  $\theta$ , between the uniaxial tensile loading direction (LD) and the normal direction (ND) of the plate. Also, different grain sizes are produced for each  $\theta$  through isochronal annealings of 96 hours at various temperatures. The conclusions are as follows.

1. The as-received grain size of 26 $\mu\text{m}$  increases systematically to 78 $\mu\text{m}$  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 the activation energy of 256 kJ/mol.

2. The 0.002 offset yield strengths are measured for four different grain sizes ( $D = 26, 43, 63, \text{ and } 78\mu\text{m}$ ) at five different tensile LDs ( $\theta = 0, 22.5, 45, 67.5, \text{ and } 90^\circ$ ) and Hall-Petch (H-P) relationships are established as a function of  $\theta$ . The measured H-P parameters of friction stress,  $\sigma_o$ , and grain size sensitivity,  $k_\sigma$ , vary significantly depending on the loading direction.

3. The  $\sigma_o$  is calculated as a function of  $\theta$  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  $\sigma_o$  as a function of  $\theta$  clearly identify changes in the dominant deformation mechanism at yielding. The extension twinning is dominant at yielding for  $\theta=0^\circ$  to  $39^\circ$ , basal slip from  $39^\circ$  to  $83^\circ$ , and prismatic slip from  $83^\circ$  to  $90^\circ$ .

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  $\theta$ , 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  $90^\circ$  with a small grain size, while yield strength will be the lowest under tension along  $45^\circ$  with a large grain size.

6. The H-P relationships for individual deformation mechanisms are identified. The grain size sensitivity  $k_\tau^\beta$  (MPa $\cdot$ mm $^{1/2}$ ) increases from basal (2.61), prismatic (5.54) to pyramidal (9.86); which is consistent with the increase in the CRSS,  $\tau_o^\beta$ , 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  $\tau_o$  (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.

## Chapter 4

### **Interplay between Texture, loading Path, and Active Deformation Mechanisms in a Wrought Mg alloy: Diffraction Measurements and Visco-plastic Self-consistent Modeling**

The effects of texture and loading paths on yielding and hardening behaviors of a wrought Mg alloy during uniaxial tensile and compressive deformation at room temperature were studied using synchrotron x-ray diffraction (S-XRD) measurements and visco-plastic self-consistent (VPSC) modeling simulation. Various initial textures were facilitated by changing the loading directions (LDs) with respect to the normal direction of the rolled plate. The critical resolved shear stresses,  $\tau_o$ , for various deformation modes used in the current VPSC model were obtained from a series of Hall-Petch relationship studies. The macroscopic stress-strain behaviors for five tensile LDs and three compression LDs were successfully simulated using a single set of constitutive properties with only the latent hardening parameter,  $h^{twin,slip}$  differing between the tensile and compression cases. The VPSC results illustrate evolutions of active deformation mechanisms, the twin volume fraction, and the texture as a function of applied strain under various loading paths. The evolutions of texture and twin volume fractions were also measured using S-XRD and the results showed good agreements with those of the VPSC model providing additional validation. The changes in dominant deformation mechanisms, their interplay with texture and loading paths, and, finally, their influence

on the hardening behavior are discussed. In this study, the  $\tau_o$  for various deformation modes as the VPSC input parameter were obtained through the Hall-Petch studied and the one set of input hardening behaviors were used to achieved the stress-strain behaviors and texture evolutions, which fit the experimental results under different loading paths and loading orientations.

#### **4.1 Introduction**

Wrought magnesium alloys exhibit significant plastic anisotropy at ambient temperatures, which limits the formability and broad applications as a lightweight structural metal [1]. The activation of a particular plastic deformation mechanism is highly dependent on the relationship between the mechanical loading direction and texture for Mg alloys with the hexagonal-close-packed (hcp) crystal structure. Various theories and modeling methods have been developed to predict the plastic anisotropy and texture evolutions by simulating the yielding and hardening characteristics and crystal reorientations at the grain level. Examples include the early Taylor-Bishop-Hill [130] and Sachs [131] theories, the self-consistent plasticity simulations [132], and finite element models [134]. The micromechanical self-consistent methods provide more accurate predictions of stress and texture developments in the low-symmetry crystal structures such as hcp alloys, compared to the Taylor's or Sach's approaches; and demand relatively lower computational costs over the finite element methods. The distinction of a crystal plasticity model comes from the method used to describe the interaction between the grains and their neighbors. The self-consistent plasticity simulation assumes the

surroundings of a grain as a homogenous effective medium (HEM). The interaction between each grain (represented by an ellipsoidal inclusion) and the HEM can be simplified to various interaction equations according to the Eshelby theory [182]. Typically, the self-consistent plasticity model provides strain responses, individual slip activities, and reorientation of each grain [137].

Recently, the anisotropic behavior and texture evolution during the deformation of hcp metals such as Mg [183] and Zr [149] have been intensively studied using the visco-plastic self-consistent (VPSC) model developed by Lebensohn *et al.* [132]. Subsequently, the elasto-plastic self-consistent (EPSC) polycrystal model was developed by Turner *et al.* [136] to better understand the elastic deformation and internal stress evolutions, which has been validated extensively using the lattice strains measured using neutron diffraction for hcp crystals such as Mg alloys [50, 137]. However, the EPSC model is not suitable for simulations of the crystal reorientation (e.g., texture evolution or twin activation) or strain rate-sensitive behaviors (e.g., stress relaxation or creep behavior). More recently, the elasto-viscoplastic self-consistent model (EVPSC) developed by Wang *et al.* [138] successfully simulated the lattice strain and texture evolutions during the uniaxial tension and compression of an AZ31 Mg alloy under large applied strains.

The single-crystal yielding and hardening parameters for the AZ31B Mg alloy reported in the literature vary widely and seem to be highly dependent on the sample process conditions and test conditions. The crystal plasticity parameters should be dependent only on the alloy composition but not on the processing or test conditions at a

given testing temperature. For example,  $\tau_o^\alpha$  (termed “initial” CRSS in most of the VPSC modeling studies) determines the shear yield stress for a specific deformation mode,  $\alpha$ , which is strongly dependent on the grain size. Recently, the authors (Wang and Choo) obtained “true”  $\tau_o$  for different deformation modes active in the AZ31B Mg alloy through Hall-Petch (H-P) relationship studies based on the dislocation pile-up theory [184]. Therefore,  $\tau_o$  in the VPSC model for a given grain size can be obtained based on the H-P parameters and the effect of loading paths (angles and directions) on the yielding behavior can be simulated accurately for different dominant deformation mechanisms and crystal orientation factors.

By using the VPSC simulation, Agnew [160] and Jain [35, 89] *et al.* showed the activation of various deformation mechanisms as a function of the initial texture during the uniaxial compression along transverse direction (TD), rolling direction (RD), and normal direction (ND) at room temperature as well as at high temperatures up to 200°C. The dominant deformation modes reported for compression along ND and compression along TD (or RD) cases were prismatic slip and extension twin, respectively. Also, Yi *et al.* [93] reported VPSC simulation results for the compression and tension cases along 0, 45, and 90 degrees to the extrusion direction of an AZ31 alloy. However, most of the simulation studies did not include a case dominated by the basal slip and, hence, the corresponding plasticity parameters have not been rigorously validated. The systematic changes in texture to activate all possible deformation modes (i.e., basal, prismatic, and pyramidal slip as well as extension twin in AZ31B Mg alloy at room temperature) with

various proportional dominances are important to establish accurate crystal plasticity parameters for a reliable simulation of plastic anisotropy.

The interactions among active deformation mode, grain orientation, and loading path become even more complex for the hardening behavior, which often justify the VPSC modeling development. For example, the changes in the hardening characteristics with the changes in the relationship between the initial texture and loading direction were shown in [94]. The sigmoidal hardening behavior is commonly shown when the extension twin predominantly accommodates the strain, while the concave down hardening curve is shown for slip dominant cases [42]. For the slip dominant deformation, the Kocks-Mecking (K-M) model has been successfully employed to describe the work-hardening behavior for several metals and alloys including aluminum, copper, titanium, and austenitic stainless steel [185]. It was also reported that the work hardening behavior can be investigated using the K-M model for AZ31B and AM60 Mg alloys processed by equal channel angular pressing [96]. According to the K-M model, the hardening behavior is highly dependent on the crystal orientation factor and dislocation characteristics.

It has also been shown that the texture alters significantly upon the activation of extension twin, which changes the crystal orientation relationship to the loading paths and subsequently deformation modes within the twinned region [33]. Such interplay between the texture and active deformation modes and its influence on the hardening behaviors can be studied using the VPSC modeling by systematically changing the twin activation and its variant selections.

In summary, the main objectives of the current VPSC work are: (1) to use “true” CRSS obtained from Hall-Petch relationship studies for accurate simulation of yielding behavior and onset(s) of various deformation mechanisms during subsequent flow behavior and (2) to use a single set of crystal plasticity parameters to simulate multiple loading orientations for both tensile and compressive deformations for a rigorous validation against varying proportional combinations of numerous dominant deformation mechanisms and their active variants.

In this chapter, we will present: (1) macroscopic stress-strain behaviors during tensile or compression deformation with loading direction,  $\theta$ , changing systematically with respect to the ND of the plate, (2) relative activities of various deformation mechanisms simulated by the VPSC model as a function of the applied strain at multiple loading orientations with respect to the initial texture; (3) corresponding texture and twin volume fraction evolutions both measured by S-XRD and modeled by VPSC; and (4) the optimized single-crystal yielding and hardening parameters for basal, prismatic, and pyramidal slip as well as extension twin. We will also discuss: (1) the effect of the loading path (loading angles and directions) on the active deformation mechanisms and texture development; (2) the interplay between extension twin, grain reorientation, and subsequent slip activities, and (3) the effect of texture and deformation mode on the macroscopic hardening behavior.



## 4.2 Experimental details

### 4.2.1 *Materials*

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 dimensions of the rolled plate are 400×250×90mm<sup>3</sup>, which allowed a series of tension and compression tests at various loading angles between normal and transverse directions of the plate. Throughout the chapter, ND, TD, RD, and LD refer to normal, transverse, rolling, and (tensile or compressive) loading direction, respectively. The average grain size is 26 ( $\pm 3.5$ )  $\mu\text{m}$ , Figure 4-1a. The average grain sizes measured using optical microscopy on ND, TD, and, RD cross sections showed similar equiaxed grain morphology [184]. The rolled Mg plate has a strong plane texture with most of the *c*-axes of the crystals aligned near parallel to the ND of the plate as shown in pole figures measured using synchrotron x-ray diffraction (S-XRD), Figure 4-1b.

### 4.2.2 *Mechanical testing*

A series of dog-bone tensile specimens (with gauge dimension of 3.0 mm wide, 1.6 mm thick, and 16.5 mm long) and cylindrical compression specimens (with dimension of 8mm long and 5mm diameter) were prepared from the rolled Mg plate by electrical discharge machining (EDM) with the angle,  $\theta$ , between LD and ND systematically changing from 0° to 90°.

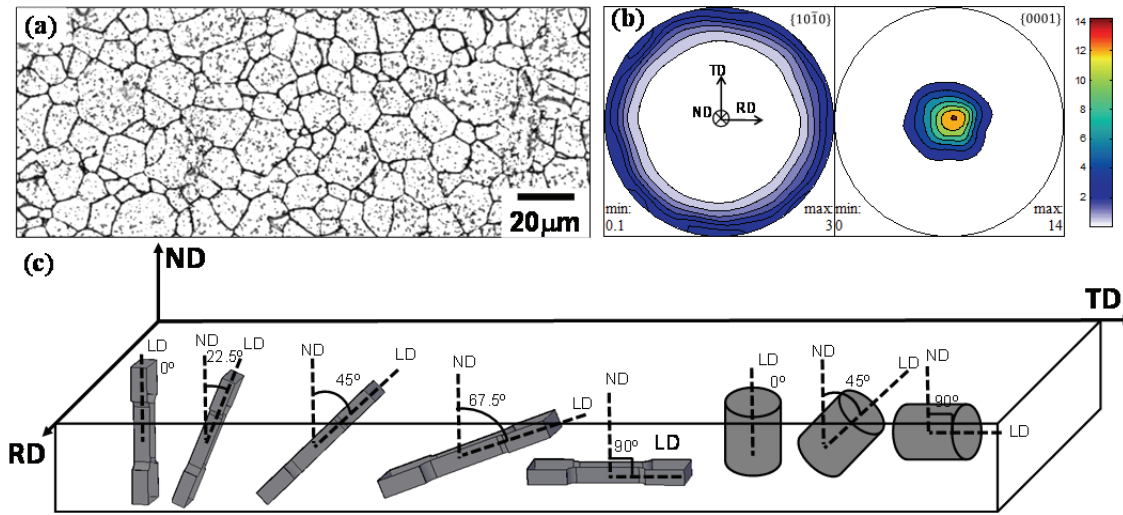


Figure 4-1. Microstructure of as-received AZ31B Mg alloy rolled plate and a schematic showing the preparation of tensile and compression specimens: (a) optical micrograph showing initial grain size ( $26\mu\text{m}\pm 3.5$ ) measured from the ND surface, (b)  $\{10\bar{1}0\}$  and  $\{0001\}$  pole figures (PFs) measured using S-XRD, and (c) a total of five different orientations were prepared for the tensile test with the angle,  $\theta_T$ , between the LD and ND ranging from  $0^\circ$ ,  $22.5^\circ$ ,  $45^\circ$ ,  $67.5^\circ$ , to  $90^\circ$ ; and three different orientations were prepared for the compression test with the angle,  $\theta_c$ , from  $0^\circ$ ,  $45^\circ$ , to  $90^\circ$ . ND, TD, RD, and LD refer to normal, transverse, rolling, and loading direction (tensile or compressive), respectively.

A total of five different orientations for the tensile tests ( $\theta_T = 0^\circ, 22.5^\circ, 45^\circ, 67.5^\circ$ , and  $90^\circ$ ) and three orientations for the compression tests ( $\theta_C = 0^\circ, 45^\circ$ , and  $90^\circ$ ) were prepared as illustrated in Figure 4-1c. For example,  $\theta_T = 0^\circ$  case is where the tensile loading orientation is along the ND of the plate, while  $\theta_T = 90^\circ$  case is where the tensile loading direction is along TD (i.e.,  $90^\circ$  to the ND). Therefore, the initial textures of the mechanical testing specimens are systematically changed with respect to the LD. All tests were performed using Instron high-resolution load frame to accurately measure the yield strengths as low as 40MPa. The load frame offers good force accuracy ( $\pm 0.5\%$ ), speed accuracy ( $\pm 1.0\%$ ), and position accuracy ( $\pm 0.5\%$ ). The initial strain rate for the deformation was  $10^{-3}\text{s}^{-1}$ . Several repeat measurements were performed for all loading paths and they showed a good reproducibility with a variation in the flow stress within  $\pm 0.5\%$ .

#### **4.2.3 High energy synchrotron x-ray diffraction**

The texture measurements were performed using synchrotron x-ray diffraction (S-XRD) at the beamline 11 ID-C at Advanced Photon Source (APS), Argonne National Laboratory. For each tensile orientation, a total of 9 samples were prepared to conduct post-mortem texture measurements at the strain levels of 0.005, 0.02, 0.03, 0.05, 0.075, 0.103, 0.127, 0.150, and 0.240. For the compression case, a total of 7 samples for the  $90^\circ$  case were prepared at the strain levels of 0.005, 0.02, 0.03, 0.05, 0.075, 0.103, and 0.150. The S-XRD texture samples were machined from the gauge section of deformed samples with the cross section of  $3\times 3\text{mm}^2$ . The beam size was  $600\times 600\mu\text{m}^2$ , the wavelength was

0.107841Å, and the nominal distance between the sample and the 2D detector was set at 1540 mm, which provided 22 reflections for the hcp Mg (from 01.0 to 03.2). 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  $\omega$  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 analysis 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 [166] for texture presentation in terms of pole figures. Further details of the texture measurements can be found in [167].

### **4.3 Visco-plastic self-consistent modeling approach**

The visco-plastic self-consistent (VPSC) model provides a detailed description of the anisotropic plastic behavior of Mg alloys. Specifically, the stress-strain behavior, texture evolution, activation and interaction of various deformation mechanisms, and the evolution of twin volume fraction can be simulated. In the VPSC model, each grain is described as an ellipsoidal inclusion with a distinct crystal orientation embedded in a homogeneous effective medium (HEM) [186, 187]. The Eshelby inclusion formalism is used to describe the interaction between each grain and the HEM. During each deformation step, the single crystal constitutive behavior describes the grain-level response and the self-consistency criteria are solved simultaneously, which maintains the

grain-level stresses and strain rates to be consistent with the boundary conditions imposed on the surrounding polycrystalline aggregates. Different stiffnesses for the grain-matrix interaction associated with the various self-consistent schemes (SCS) can be chosen for the linearization. In the present study, the *Tangent* SCS was used, which represent the lower boundaries of the interaction. Overall, *Tangent* SCS provides the reasonable stress-strain behaviors and R values prediction in Mg with slight off in texture evolution.

Plastic deformation of a crystal is facilitated by various crystallographic slip and twinning systems  $\alpha$  with an orientation definition  $(\mathbf{s}^\alpha, \mathbf{n}^\alpha)$ . Here,  $\mathbf{s}^\alpha$  and  $\mathbf{n}^\alpha$  are the slip/twin direction and normal direction of the slip/twin system  $\alpha$ , respectively. Four deformation systems are considered to accommodate the plastic deformation at room temperature:  $\{0001\} < 11\bar{2}0 >$  basal slip;  $\{\bar{1}010\} < 11\bar{2}0 >$  prismatic slip;  $\{10\bar{1}1\} < 11\bar{2}3 >$  pyramidal slip; and  $\{10\bar{1}2\} < \bar{1}011 >$  extension twin. Each deformation system is defined using a set of single-crystal plasticity parameters. Voce hardening law is employed to describe the hardening responses of each deformation mechanism using instantaneous critical resolved shear stress (CRSS),  $\hat{\tau}^\alpha$ , as a function of accumulated shear strain in the grain,  $\gamma_{ac}$ :

$$\hat{\tau}^\alpha = \tau_o^\alpha + (\tau_1^\alpha + h_1^\alpha \gamma_{ac}) \left[ 1 - \exp\left(-\frac{h_o^\alpha}{\tau_1^\alpha} \gamma_{ac}\right) \right] \quad 4-1$$

where  $\tau_o$ ,  $h_o$ ,  $h_1$ , and  $\tau_o + \tau_1$  are the initial CRSS, the initial hardening rate, asymptotic hardening rate, and back-extrapolated CRSS, respectively.

The grain (crystal) level plastic strain rate  $\mathbf{d}^g$  is:

$$\mathbf{d}^g = \sum_{\alpha} \dot{\gamma}^\alpha \mathbf{P}^\alpha \quad 4-2$$

where  $\dot{\gamma}^\alpha$  is the shear rate of slip (twin) system  $\alpha$ , and  $\mathbf{P}^\alpha$  is the associated Schmid tensor:

$$\mathbf{P}^\alpha = \frac{1}{2} (\mathbf{s}^\alpha \mathbf{n}^\alpha + \mathbf{n}^\alpha \mathbf{s}^\alpha) \quad 4-3$$

For slip,

$$\dot{\gamma}^\alpha = \dot{\gamma}_o \left| \frac{\tau^\alpha}{\tau_{CR}^\alpha} \right|^{1/m} \text{sgn}(\tau^\alpha) \quad 4-4$$

For twin,

$$\dot{\gamma}^\alpha = \begin{cases} \dot{\gamma}_{0o} \left| \frac{\tau^\alpha}{\tau_{CR}^\alpha} \right|^{1/m} & \text{for } \tau^\alpha > 0 \\ 0 & \text{for } \tau^\alpha \leq 0 \end{cases} \quad 4-5$$

,where  $\dot{\gamma}_o$  is a reference value of slip/twin rate,  $m$  is the slip/twin rate sensitivity, and  $\tau^\alpha$  is the resolved shear stress:

$$\tau^\alpha = \boldsymbol{\sigma}^g : \mathbf{P}^\alpha \quad 4-6$$

$\tau_{CR}^\alpha$  is the critical resolved shear stress (CRSS) and sgn is the sine function.

The evolution of  $\tau_{CR}^\alpha$  is taken in the form of

$$\dot{\tau}_{CR}^\alpha = \frac{d\hat{\tau}^\alpha}{d\gamma_{ac}} \sum_{\beta} h^{\alpha\beta} \dot{\gamma}^\beta \quad 4-7$$

where  $\gamma_{ac} = \sum_{\alpha} |\gamma^\alpha|$  is the accumulated shear strain in the grain,  $h^{\alpha\beta}$  is the latent hardening coupling coefficient which empirically accounts for the obstacles on system  $\alpha$  associated with system  $\beta$ .  $\hat{\tau}^\alpha$  is the threshold stress and is characterized by Equation (1).

The contribution of the extension twin to the grain reorientation was represented by the predominant twin reorientation (PTR) scheme developed by Tomé *et al.* [135, 145]. Within each grain  $g$ , the PTR scheme tracks the shear strain  $\gamma^{\alpha,g}$  contributed by

each twin system  $\alpha$ , and the associated volume fraction  $V^{\alpha,g} = \gamma^{\alpha,g} / \gamma^{tw}$ . Here  $\gamma^{tw}$  is the characteristic shear (constant) associated with the twin. Crystallographically equivalent twins belong to the same twin mode. For example, the  $\{10\bar{1}2\}$  twin systems, which are activated by  $c$ -axis tension, constitute the tensile twin mode. The sum of  $V^{\alpha,g}$  over all twin systems associated with a given twin mode over all grains represents the “accumulated twin fraction”,  $V^{acc,mode}$ .

$$V^{acc,mode} = \sum_g \sum_{\alpha} V^{\alpha,g} \quad 4-8$$

In the PTR scheme, after each deformation increment, a grain is randomly selected to check if the predominant twinning system exceeds a “threshold value”. If so, the grain is entirely reoriented to a new orientation according to the predominant twinning system. The volume fraction of this reoriented grain is added to the so-called “effective twin fraction”,  $V^{eff,mode}$ . The process is repeated until either all grains are checked or the effective twin fraction exceeds the accumulated twin fraction. In the latter case, the reorientation by twinning is ceased and the next deformation step is processed. The aforementioned threshold value is defined as

$$V^{th,mode} = A^{th1} + A^{th2} \frac{V^{eff,mode}}{V^{acc,mode}} \quad 4-9$$

,where  $A^{th1}$  and  $A^{th2}$  are two material constants. This approach statistically solves the practical problem that the number of twin orientations would grow continually if each activated twinning system was represented by a new orientation. Additionally, it maintains the twinned volume fraction at a level that is consistent with the shear activity of the twins contributing to the deformation.

Finally, the yielding and hardening “input” parameters ( $\tau_0^\alpha$ ,  $h_0^\alpha$ ,  $h_1^\alpha$ ,  $\tau_0^\alpha + \tau_1^\alpha$ , and  $h^{\alpha\beta}$ ) used in the current study are listed in Table 4-1, which are also one of the main outcomes since the simulation process is iterative.

As mentioned earlier,  $\tau_0^\alpha$  for various deformation modes were obtained from the Hall-Petch relationship studies [184]. Two different sets of latent hardening parameters were used for the tension and compression cases, while keeping other parameters constant for all cases. The initial crystal orientation matrix was obtained from the measured texture of the as-received specimen, Figure 4-1b. For all cases, 16,240 grains (i.e., orientations) were modeled.

## 4.4 Results

### 4.4.1 *Macroscopic stress-strain behavior as a function of the loading angle, $\theta$*

#### 4.4.1.1 Tensile behavior

The tensile stress-strain curves measured at five different loading orientations are presented in Figure 4-2a. The open symbols in Figure 4-2(b-f) are the VPSC simulation results shown in comparison to the measured data for each orientation. The comparison shows good agreements for all five tensile orientations, with only a slight mismatch between the strain of 0.100 and 0.125 for the  $\theta_T = 0^\circ$  case.

The different yielding and hardening behaviors indicate that the dominant deformation mechanisms are different for different  $\theta_T$  cases. The 0.002 yield stress of the  $0^\circ$  case (Figure 4-2b) is much lower (about 52MPa) than that of the  $90^\circ$  case (about 120MPa).



Table 4-1. Critical resolved shear stress (CRSS) and hardening parameters used for the VPSC modeling.  $\tau_o$ ,  $h_o$ ,  $h_1$ , and  $\tau_o + \tau_1$  are CRSS, initial hardening rate, asymptotic hardening rate, and back-extrapolated CRSS, respectively.  $h^*$  is the latent hardening parameter, and B, Pr, Py, and T represent basal, prismatic, pyramidal slip and extension twin, respectively. Two different sets of latent hardening parameters were used for the tension and compression (in parentheses) cases. For example,  $h^*B$  of 1.2 for the twin is the  $h^{\alpha,\beta}$  term in equation 4-7 expressing the hardening of basal slip imposed by the extension twin.

| <b>Deformation modes, <math>\alpha</math></b> | <b><math>\tau_o</math></b> | <b><math>\tau_1</math></b> | <b><math>h_o</math></b> | <b><math>h_1</math></b> | <b><math>h^*B</math></b> | <b><math>h^*Pr</math></b> | <b><math>h^*Py</math></b> | <b><math>h^*T</math></b> |
|-----------------------------------------------|----------------------------|----------------------------|-------------------------|-------------------------|--------------------------|---------------------------|---------------------------|--------------------------|
| Basal slip <a>                                | 24                         | 4                          | 2550                    | 70                      | 1                        | 1                         | 1                         | 1                        |
| Prismatic slip <a>                            | 52                         | 27                         | 2700                    | 300                     | 1                        | 1                         | 1                         | 1                        |
| Pyramidal slip <c+a>                          | 90                         | 80                         | 4000                    | 62                      | 1                        | 1                         | 1                         | 1                        |
| Extension Twin                                | 29                         | 0                          | 0                       | 0                       | 1.2 (0.6)                | 1.4 (1.8)                 | 1.0 (0.8)                 | 1                        |

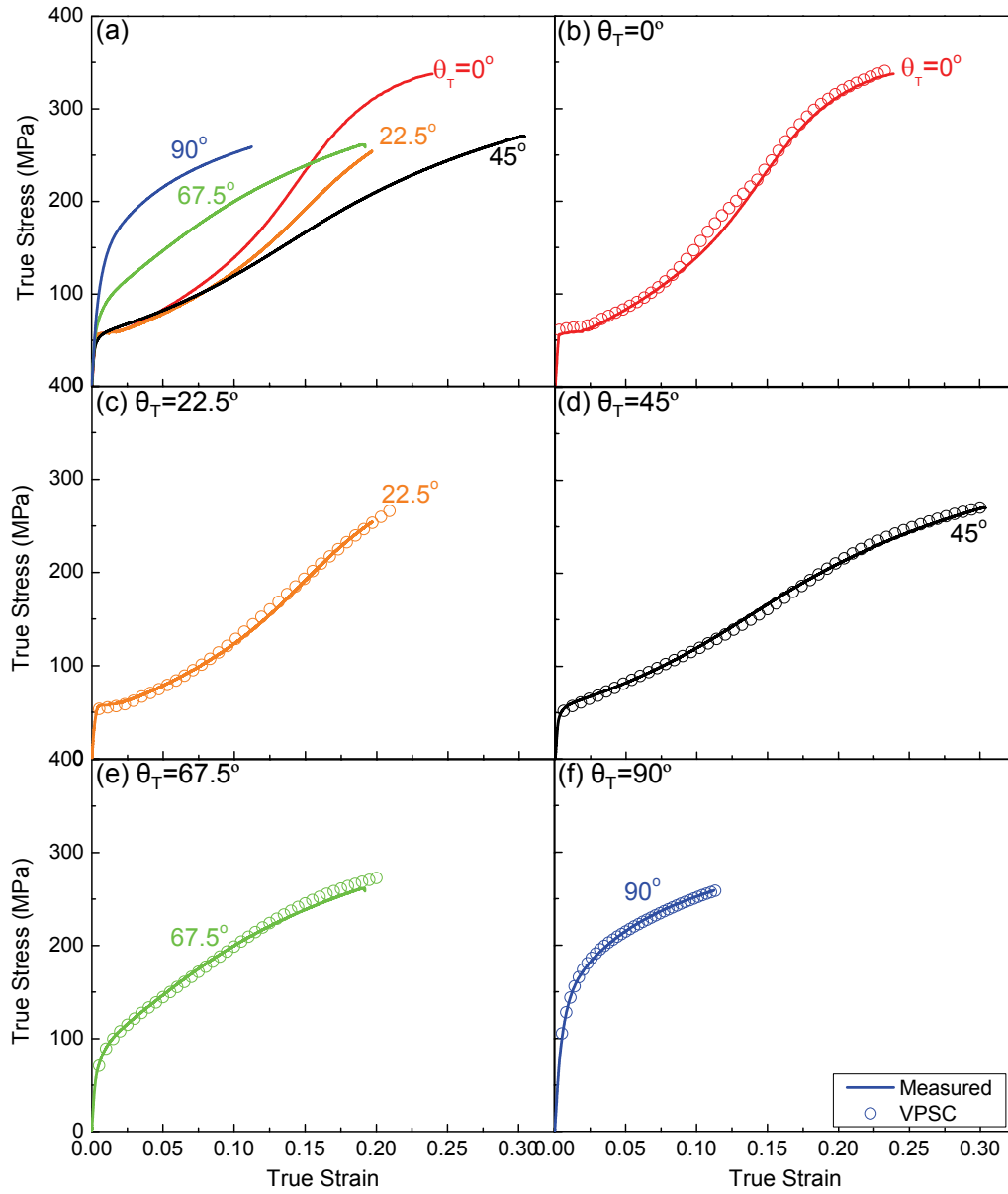


Figure 4-2. Tensile stress-strain curves measured and simulated at five different loading orientations,  $\theta_T$ . The lines are measured data and the symbols are simulation results from the VPSC model. (a) Measured tensile stress-strain curves for all five orientations, (b)  $\theta_T=0^\circ$ , (c)  $\theta_T=22.5^\circ$ , (d)  $\theta_T=45^\circ$ , (e)  $\theta_T=67.5^\circ$ , and (f)  $\theta_T=90^\circ$ .

After yielding, the flow curve of the  $0^\circ$  case exhibits a plateau until 0.020 strain followed by a sigmoidal hardening behavior, which implies profuse extension twinning. This behavior is similar to the compression along RD (or TD) of hot-rolled plates or compression along the extrusion direction (ED) of a hot-extruded specimens [137, 188]. For the  $22.5^\circ$  case (Figure 4-2c), the yield strength (50MPa) is almost identical to that of the  $0^\circ$  case, followed by a plateau and a concave-up hardening curve with a slightly lower hardening rate. The  $45^\circ$  case (Figure 4-2d) shows similar yield strength (45MPa) as the  $0^\circ$  or  $22.5^\circ$  cases, but the hardening rate does not increase as much, suggesting a more slip-dominant case. The  $45^\circ$  case shows the largest ductility among all conditions tested. On the other hand, the yield strengths of the  $67.5^\circ$  (72MPa) and  $90^\circ$  (120MPa) cases are much higher, followed by concave-down parabolic flow curves, Figure 4-2(e-f).

#### 4.4.1.2 Compressive behavior

Figure 4-3 shows the compressive stress-strain curves measured (lines) and simulated (symbols) at  $\theta_C = 0^\circ$ ,  $45^\circ$  and  $90^\circ$ . The results show good agreements for the compression cases as well, where significant variations in the yielding and hardening behaviors are evident with the changes in the loading angle,  $\theta_C$ .

The concave-down flow curve is observed for the  $0^\circ$  cases with a high yield stress, while the concave-up behavior is observed for the  $90^\circ$  case with much lower yield stress. This is qualitatively opposite to the tensile cases. For  $\theta_C=0^\circ$ , the specimen yields at 116MPa with a high initial hardening rate up to 350MPa, where it decreases sharply. The fracture strain of the  $0^\circ$  case is small at about 0.10.

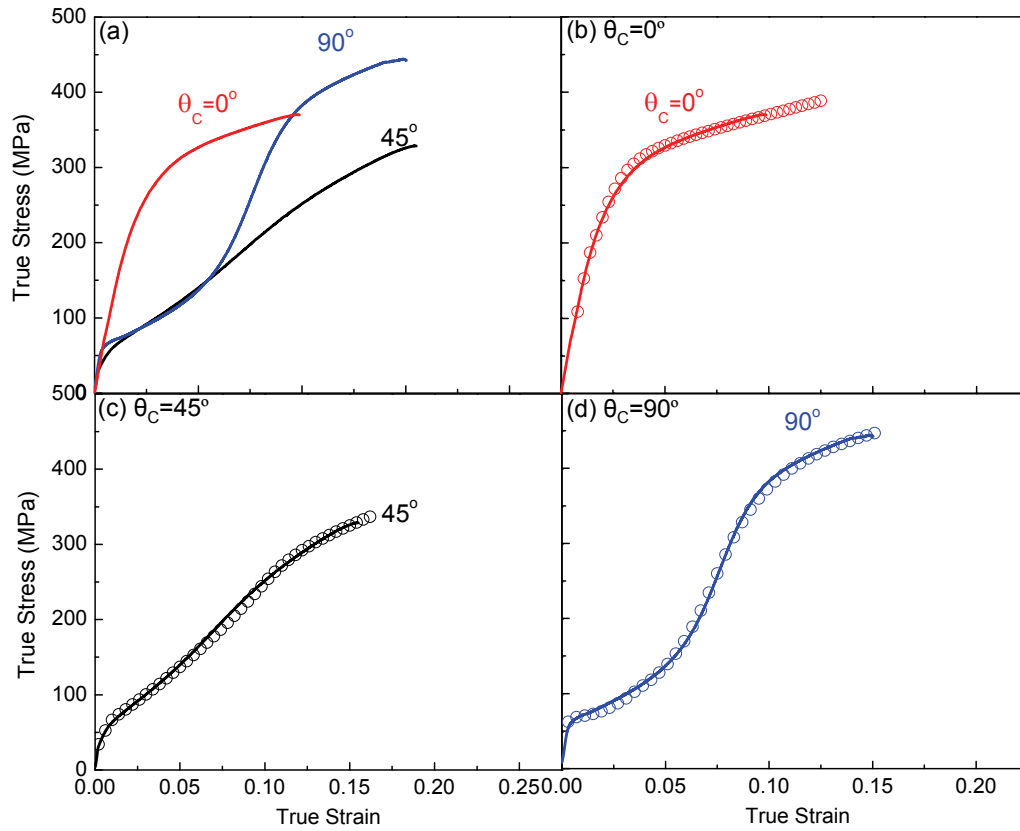


Figure 4-3. Compressive stress-strain curves measured and simulated at three different loading orientations,  $\theta_C$ : (a) all three compressive stress-strain curves obtained from experiments, (b)  $\theta_C=0^\circ$ , (c)  $\theta_C=45^\circ$ , and (d)  $\theta_C=90^\circ$ . The lines are measured data and the symbols are simulation results from the VPSC model.

The yield strengths of the 45° (46MPa) and 90° (63MPa) cases and the initial hardening rates (up to about 0.06 strain) are lower than those of the 0° case. However, the hardening rate of the 90° case increases sharply at about 0.06 strain and then gradually decreases beyond 0.075 strain. The maximum flow stress of the 90° case (445MPa) is the highest among all cases tested.

In summary, the anisotropic tensile and compressive behaviors, clearly manifested by the variations in the yielding and hardening behaviors, indicate that different deformation modes are activated with the changes in  $\theta$  and their relative dominances evolve systematically with the applied strain. The good agreements between the measured and simulated stress-strain curves for all eight loading paths provide ample validation of the current VPSC modeling results.

#### ***4.4.2 Active deformation mechanisms as a function of the loading angle, $\theta$***

##### ***4.4.2.1 Tension cases***

The evolutions in relative activities of various deformation mechanisms and the twin volume fraction are shown as a function of strain for 0°, 22.5°, 45°, 67.5°, and 90° cases in Figure 4-4. The solid lines represent the activities with the matrix (i.e., parent grains), while the dashed lines represent those within the twinned daughter grains. The relative activity of a particular deformation mode,  $\alpha$ , represents the percent of the plastic aggregated strain accommodate by  $\alpha$  in each macrostrain accumulation step.

In the  $\theta_1=0^\circ$  case (Figure 4-4a), the plastic deformation initiates with the extension twin.

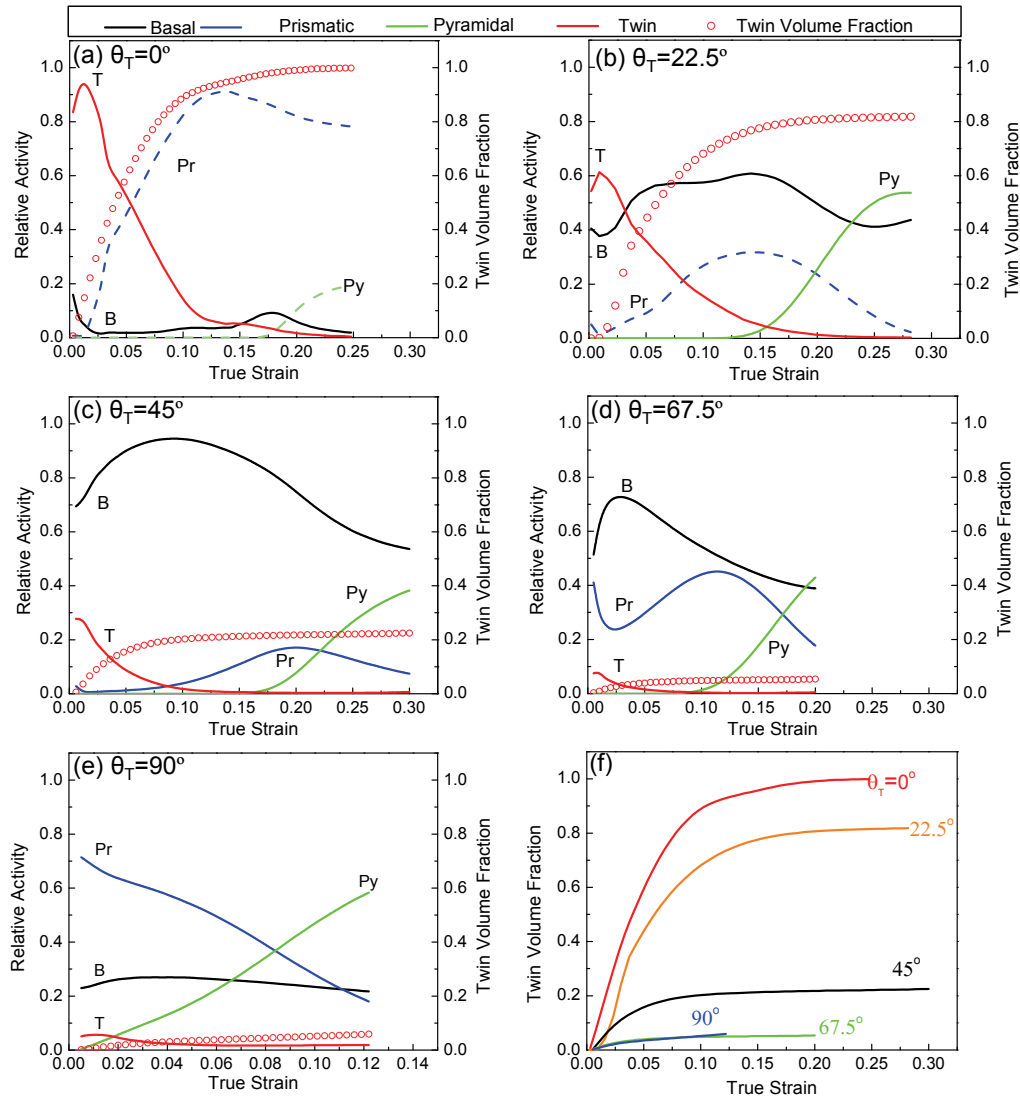


Figure 4-4. Relative Relative activities of various deformation mechanisms and the twin volume fraction evolution as a function of applied tensile strain at five different loading angles,  $\theta_T$ : (a)  $\theta_T = 0^\circ$  loading case, (b)  $22.5^\circ$ , (c)  $45^\circ$ , (d)  $67.5^\circ$ , (e)  $90^\circ$ , and (f) comparison of twin volume fraction evolutions for all five tensile orientations. The dashed line represents slip activities within twinned daughters, while solid lines indicate the slip activities occurring either within the parent or within both the parent and daughter.

With the increase in the applied strain, the twin volume fraction increases rapidly to about 55% at 0.125 strain, which means almost all of the parent grains re-orientated at this point. The relative activity of prismatic slip increases concurrently with the increase in the twin volume fraction. At the strain of 0.125, the relative activity of prismatic slip is 0.90, which is the single dominant deformation mechanism at this point. When the twinned grains reorient, the prismatic slip becomes dominant deformation mechanism within the twinned daughter grains. More detailed discussion on the locations of slip activities (i.e., within either the parent or daughter), the interaction between slip and twin, and their influence on the hardening behavior will be presented later in Section 3.5.5. At higher strains beyond 0.15, the basal and pyramidal slips are activated. The basal slip provides very limited strain with maximum relative activity less than 0.1.

In the 22.5° case (Figure 4-4b), the active deformation mechanisms at yielding are a combination of basal slip and extension twin. The activity of the extension twin decreases rapidly with the increase in the strain resulting in about 80% twin volume fraction, while the basal slip remains at the similar level. The relative activity of the prismatic slip peaks at 0.3 with the increase in the twin volume fraction when the strain is about 0.13 and then gradually replaced by the pyramidal slip. Since the basal slip occurs both in the parent and daughter grains, the solid line is used in Figure 4-4b.

In the 45° case (Figure 4-4c), the basal slip is the primary mode of plastic deformation with the relative activity no less than 0.5. At yielding, the extension twinning is the second dominant mode whose relative activity is at about 0.3. However, the activity of extension twin diminishes quickly within the strain of about 0.08 resulting in twin

volume fraction of about 20%. When the strain increases to 0.175 the pyramidal slip accommodates some strain by partially replacing the basal slip. The prismatic slip is the least active mode with the peak activity less than 0.2.

In the 67.5° case (Figure 4-4d), the basal and prismatic slips dominate the plastic deformation throughout. The pyramidal slip is activated at about 0.10 strain. The twin activity is very limited with the final volume fraction less than 5%.

In the 90° case (Figure 4-4e), the plastic deformation is initiated by the prismatic slip, which is gradually replaced by the pyramidal slip. The basal slip activity remains constant at about 0.2 throughout. The twin volume fraction stays below 5% since most of the *c*-axes are under compression.

Figure 4-4f compares the evolution of twin volume fractions as a function of strain for all five tensile orientations. Overall, the twin volume fraction increases with the increase in the applied strain. However, the maximum twin volume fractions attained systematically decreases (from 100% to less than 5%) as the loading angle  $\theta$  increases.

#### 4.4.2.2 Compression cases

Figure 4-5 presents the relative activities of various deformation modes for the compression cases. For  $\theta_c=0^\circ$  case (i.e., compression along ND), the basal slip is activated at yielding, Figure 4-5a. Then, it is rapidly replaced by the pyramidal slip. The relative activities of both basal and pyramidal slips are about 0.5 when the strain is about 0.03. Neither prismatic slip nor extension twin is activated in this case.



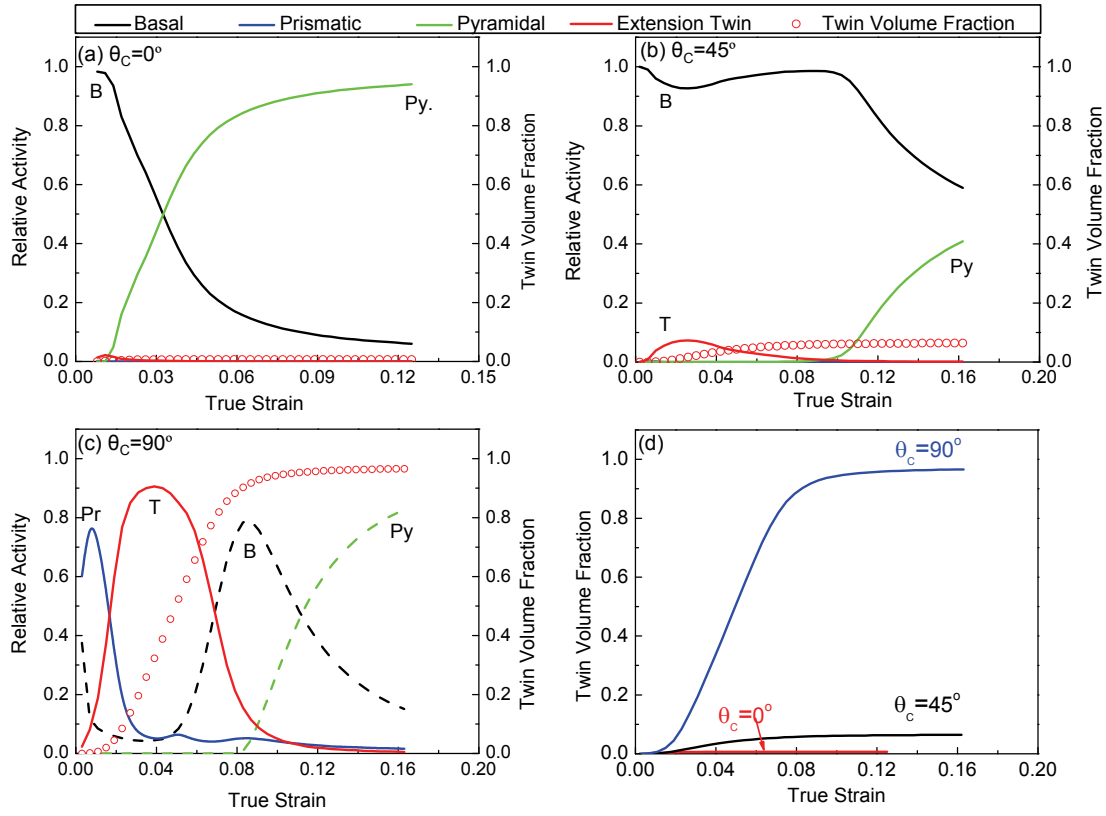


Figure 4-5. Relative activities of various deformation mechanisms and the twin volume fraction evolution as a function of applied compressive strain at three different loading angles,  $\theta_c$ : (a)  $\theta_c = 0^\circ$  loading case, (b)  $45^\circ$ , (c)  $90^\circ$ , and (d) comparison of twin volume fraction evolutions for all three compressive orientations. The dashed line represents slip activities within twinned daughters, while solid lines indicate the slip activities occurring either within the parent or within both the parent and daughter.

For the 45° case (Figure 4-5b), basal slip is the single dominant mode up to the strain of 0.10, beyond which pyramidal slip is activated. The activity of extension twin is very limited with the maximum volume fraction less than 0.10.

For the 90° case (i.e. compression along TD), the interactions among various deformation modes are rather complicated. Figure 4-5c shows that the prismatic slip occurs first ( $< 0.02$  strain), followed by the extension twin that dominates until the strain of about 0.07. With the increase in the twin fraction, the basal slip becomes active, which, in turn, is gradually replaced by the pyramidal slip.

Figure 4-5d compares the twin volume fraction evolutions with the applied strain at three compression loading orientations. The trend is opposite to that of the tension cases. For the  $\theta_C=90^\circ$  case, the twin volume fraction reaches 90% at the strain of 0.08 while that the maximum twin fraction for the 45° and 0° cases are less than 10% and 2%, respectively.

#### ***4.4.3 Texture evolution as a function of the loading angle, $\theta$***

##### ***4.4.3.1 Tension texture***

The texture evolutions in the tensile specimens were analyzed as a function of applied strain using the S-XRD measurements. The  $\{0002\}$  and  $\{10\bar{1}0\}$  pole figures (PFs) for various tensile LD ( $\theta_T$ ) cases are presented in Figure 4-6 to illustrate the texture evolution.

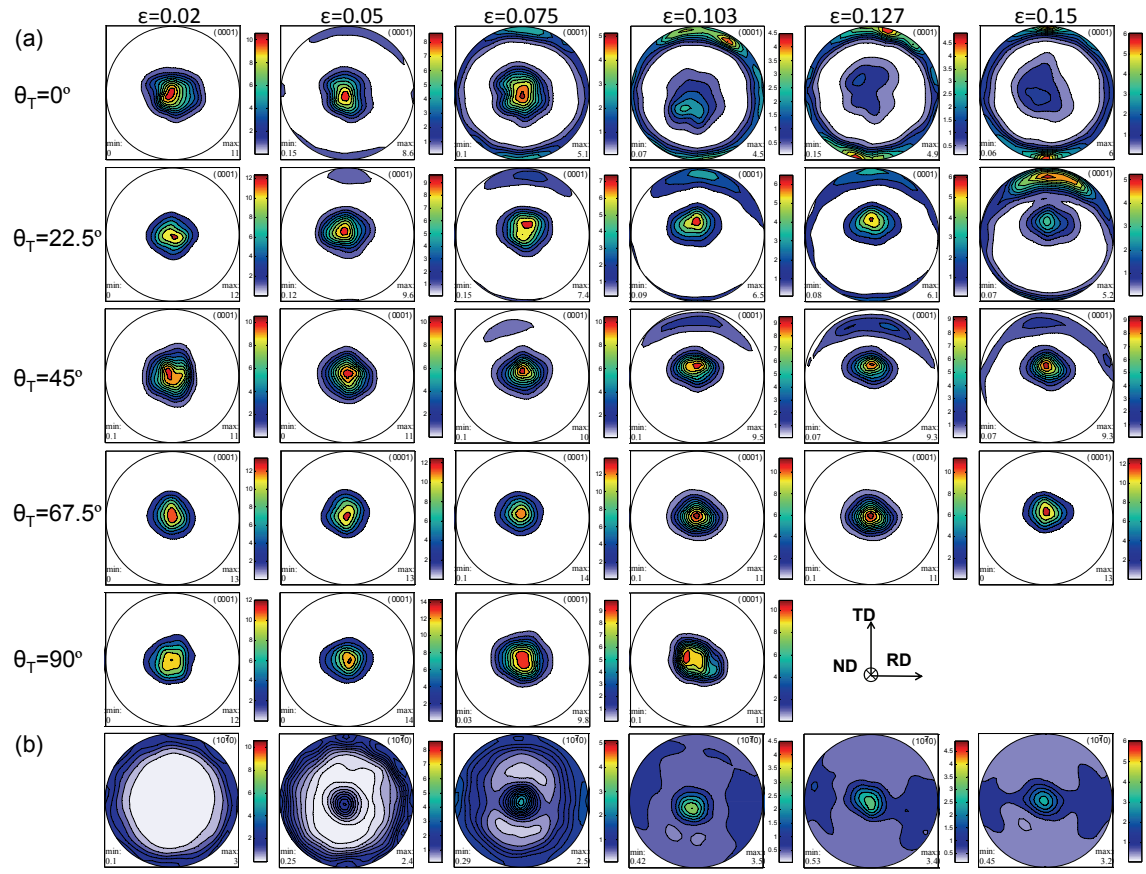


Figure 4-6.  $\{0002\}$  and  $\{10\bar{1}0\}$  pole figures (PFs) measured as a function of the applied tensile strain for the five loading orientations,  $\theta_T$ , using S-XRD: (a)  $\{0002\}$  PF and (b)  $\{10\bar{1}0\}$  PF.

The lower symmetry of the hcp crystal structure and the higher plastic anisotropy involving the twinning could cause the texture to evolve more significantly than those observed in face-center cubic (fcc) or body-center cubic (bcc) metals even under a small deformation [29]. The initial texture is the typical plane texture of the rolled Mg plate as shown in Figure 4-1b. The {0002} poles, which represent the *c*-axes of the crystals, are almost all parallel to the ND of the plate with a slight angular distribution of about 10°. The texture changes with the increase in the applied strain are highly dependent on the  $\theta_T$ .

Let us first examine the measured {0002} PFs for various  $\theta_T$  angles, Figure 4-6a. The texture changes significantly starting from a small strain ( $<0.05$ ) for the  $\theta_T=0^\circ$  case. When the strain is at 0.05, the {0002} poles near ND started to shift to the TD. With further increase in the strain, the {0002} poles shift from ND to the TD-RD plane forming a circumferential rim on the PF. When the strain increases to 0.127, the maximum intensity of the {0002} poles along ND decreased by a factor of 2.7. At the fracture strain of xxx, the {0002} intensity near ND is almost depleted and the intensity distribution around the rim (i.e., in the RD-TD plane) is rather homogeneous with the maximum intensity near TD.

At  $\theta_T = 22.5^\circ$ , the texture development shows a qualitatively similar behavior as the  $0^\circ$  case. However, the re-orientation of *c*-axes of crystals is quite asymmetric with a more pronounced development of the rim near the north pole in accordance with the changes in the tensile loading angle from the ND to  $22.5^\circ$  towards TD. . At the strain of  $0.05 \sim 0.1$ , some grains started to re-orient to the direction which is about  $75^\circ$  from north of the ND consuming the basal poles near ND but inclined about  $15^\circ$  towards the south

pole. As the applied strain continues to increase, the rim thickens with the depletion of the ND intensity.

At  $\theta_T = 45^\circ$ , the re-orientation of ND poles starts at higher strains (0.075 ~ 0.103), is more asymmetric, and less pronounced.

During the tension along  $67.5^\circ$  and  $90^\circ$ , the plane texture remains unchanged given strain range studied. With increase in the applied strain (above 0.075), the  $c$ -axis tends to spread out towards to the RD slightly. However, most of the  $c$ -axes are still aligned parallel to the ND with the texture intensity higher than 12 times the random.

The measured  $\{10\bar{1}0\}$  PF evolutions are presented in Figure 4-6b. Initially, it shows a rim in the PF, which indicates that most of the prism plane normals are perpendicular to ND and radially distributed within the TD-RD plane quite homogenously. At  $\theta_T = 0^\circ$ , it starts to re-orient from the rim to the center of the PF when the strain is at about 0.05. The intensity of the  $\{10\bar{1}0\}$  component at the center keeps increasing with the increase in strain depleting intensity around the rim. The  $\{10\bar{1}0\}$  poles also show similar evolution at  $\theta_T$  of  $22.5^\circ$  and  $45^\circ$ . The prism poles appear near ND but about  $15^\circ$  and  $35^\circ$  inclined towards TD at strains of about 0.05 and 0.075 for the  $22.5^\circ$  and  $45^\circ$  cases, respectively. The rim in the PF does not fully diminish in these two cases. For the  $67.5^\circ$  and  $90^\circ$  cases, the  $\{10\bar{1}0\}$  PFs remain unchanged with the increase in the strain.

Figure 4-7 shows the texture evolutions obtained from the VPSC simulation for the tensile deformation for the five different loading orientations.

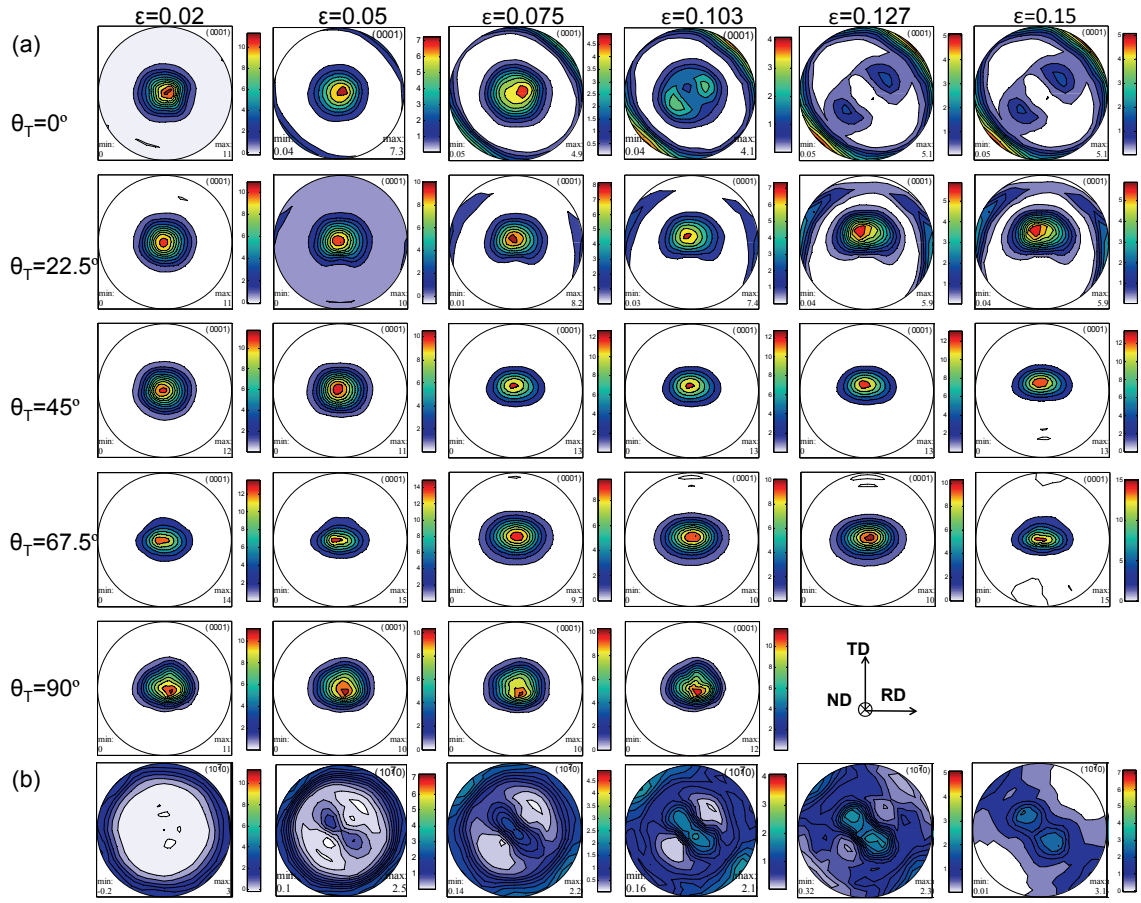


Figure 4-7. {0002} and {10 $\bar{1}$ 0} pole figures (PFs) measured as a function of the applied tensile strain for the five loading orientations,  $\theta_T$ , using VPSC: (a) {0002} PF and (b) {10 $\bar{1}$ 0} PF.

Overall, the texture evolutions show a good agreement with the PFs measured using the S-XRD (Figure 4-6), which provides a validation of the current VPSC simulation in addition to the stress-strain curves. However, there are some differences between the simulation and experimental results, especially in the  $0^\circ$ ,  $22.5^\circ$ , and  $45^\circ$  cases, where extension twinning is prevalent. In the  $0^\circ$  case, Figure 4-7a, the  $\{0002\}$  component initially appears at 0.03 strain in the TD-RD plane (i.e., on the rim of the PF), not at the north and south poles but instead with about  $30^\circ$  rotation from TD toward to the RD. With the increase in strain, the rim thickens and becomes more homogeneous. However, the ND poles re-orient predominantly towards the TD with the  $40^\circ$  clockwise rotation. In particular, this trend is quite visible at strains higher than 0.15. Such trend is the most significant qualitative difference between the measured and simulated textures and it will be discussed later in terms of the PTR scheme employed in the current VPSC simulation.

For the  $22.5^\circ$  case, Figure 4-7a, two  $\{0002\}$  components first appear on the left and right side of the rim at 0.05 strain and on the TD at 0.127 strain gradually filling up the rim asymmetrically with the increase in the. The highest intensity in the rim is near  $45^\circ$  from the TD towards RD similar to the  $0^\circ$  case. For the tension along  $45^\circ$  case, the rim is not as visible from the VPSC simulation even at the highest strain level mainly due to the difference in scale. The  $\{0002\}$  component was observed  $20^\circ$  from the ND towards TD in the VPSC simulation. Moreover, the asymmetric depletion of the  $\{0002\}$  component near ND is apparent indicating the re-orientation of this axis due to the extension twinning. The quantitative comparisons of the twin volume fraction evolutions from the VPSC and S-XRD presented later will also serve as a validation of the texture

evolutions simulated by the VPSC. The texture evolution of 67.5° and 90° cases are almost identical to ones observed from the S-XRD measurements. The differences in the texture evolutions obtained from the S-XRD and VPSC will be discussed in section 4.5.2 in terms of the PTR scheme and the SCS used in the current VPSC simulation. Finally, Figure 4-7b presents the  $\{10\bar{1}0\}$  PFs for the 0° tensile case, which shows a reasonable agreement with the measured data. It exhibits a behavior expected from the  $\{0002\}$  PF evolution discussed earlier consistent with the measured behavior.

#### 4.4.3.2 Compression texture

For the compression texture, the  $\theta_c = 0^\circ$  and  $45^\circ$  cases show very little changes with applied strain as expected from the tensile cases where only dislocation slips are dominant deformation mechanisms. Hence, only the texture evolutions obtained for the 90° compression case ( $\theta_c = 90^\circ$ ) are presented in Figure 4-8 from both measured and simulated data.

As expected from previous studies, significant texture development was observed from both methods. According to the S-XRD measured  $\{0002\}$  PFs, Figure 4-8a, the grain re-orientation is first apparent at the strain of 0.075. The  $\{0002\}$  poles re-orient from the center (ND) to the south/north poles (TD) and the intensity of the  $\{0002\}$  poles at the center continuously decreases with the increase in strain. As a result, the  $c$ -axes of the crystals re-orient from the ND to the TD during the compression along TD due to the activation of the extension twin. At the north/south pole, the intensity of  $\{0002\}$  increases until almost all of the  $\{0002\}$  components shift from the center and the



maximum intensity at the south/north pole becomes close to 11 when the strain is about 0.10 according to the S-XRD measurements. The VPSC simulated texture evolution shows onset of the  $\{0002\}$  pole re-orientation at smaller strains (at strains as low as 0.02) compared to the S-XRD measurement. In addition, the strongest intensity is shown at about  $15^\circ$  of the TD towards to the RD in the simulated results instead of the exact TD, which was the case for the experimental data. Overall, the qualitative trend is quite consistent between the two data. The  $\{10\bar{1}0\}$  component distribution shows a homogenous rim at the initial stage with the prism plane normal perpendicular to the ND. With the increase in strain, the rim of prism poles rotates  $90^\circ$  resulting in a horizontal band at the equator at high strains. Again, both measured and simulated results show good agreements.

## **4.5 Discussion**

### ***4.5.1 Crystal plasticity parameters for various deformation mechanisms***

VPSC simulation is employed to understand the effect of the changes in the initial textures on the activation of various deformation mechanisms at different stages of plastic deformation and their influence on the hardening and texture evolution. The parametric model simulations were conducted for the current study by iteratively fitting the model to room-temperature mechanical test data and texture evolutions measured for specimens with different initial textures.

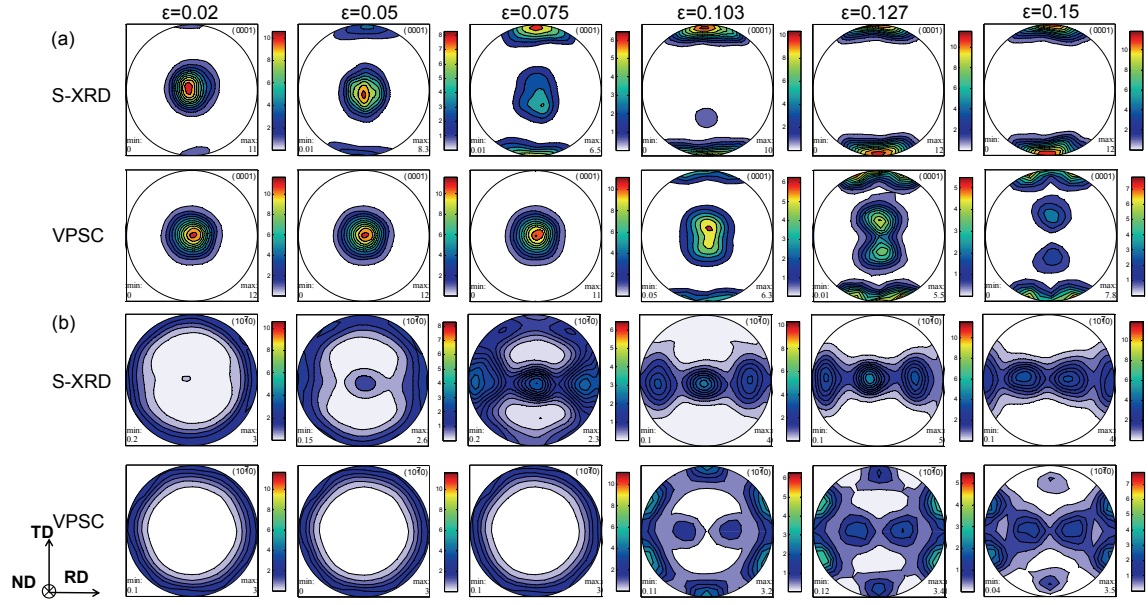


Figure 4-8. {0002} and {10 $\bar{1}$ 0} pole figures (PFs) both measured (S-XRD) and simulated (VPSC) as a function of the compressive strain at  $\theta_C = 90^\circ$ : (a) {0002} PF and (b) {10 $\bar{1}$ 0} PF. .

By using the single crystal hardening parameters as an input for tension and compression cases, Table 4-1, the simulated stress-strain behaviors and the texture evolutions showed good agreements with the characteristic features of experimental results with the different loading orientations.

The “ $\tau_0$ ” used in the current VPSC simulation is the resolved shear stress at a given grain size (26 $\mu\text{m}$ ) even though it is commonly referred to as the “initial CRSS”. Recently, Wang and Choo reported the experimentally measured Hall-Petch relationship parameters for various deformation mechanisms at ambient temperature, Table 4-2.

Therefore, the “apparent” resolved shear stresses (MPa) at grain size of 26 $\mu\text{m}$  for the basal, prismatic, pyramidal slip, and extension twinning are estimated as 26, 52, 101, and 27, respectively. These values are used as the initial input parameters for  $\tau_0$  in the current VPSC.

The hardening parameters ( $h_0^\alpha$ ,  $h_1^\alpha$ ,  $\tau_0^\alpha + \tau_1^\alpha$ ) were established from the iterative optimization through the fitting between the measured stress-strain behaviors and texture evolution. It was found that, the twin-twin interaction has to be set as 0 for  $h_0^{\text{twin}}$  and  $h_1^{\text{twin}}$  to accurately describe the plateau stage at the beginning of the plastic deformation for the twin-favorable cases. Moreover, the latent hardening parameters are tuned to match the stress-strain behaviors for all loading paths, which, in turn, provide insights to the twin-slip interactions. It is worth noting that the hardening parameters including  $\tau_0$ ,  $h_0^\alpha$ ,  $h_1^\alpha$ , and  $\tau_0^\alpha + \tau_1^\alpha$  are the same for all loading orientations for both tension and compression cases.

Table 4-2. Hall-Petch relationship parameters of various deformation mechanisms for AZ31B Mg alloy at D=26 $\mu$ m in [184].  $\tau_o$  and  $k_\tau$  are the friction stress and grain size sensitivity of the deformation mode,  $\alpha$ . “ $\tau_o$ ” in visco-plastic self-consistent (VPSC) modeling is calculated for a grain size (D) of 26 $\mu$ m

| <b>Deformation.<br/>Mode, <math>\alpha</math></b> | <b><math>\tau_o</math> (MPa)</b> | <b><math>k_\tau</math><br/>(MPa<math>\cdot</math>mm<sup>1/2</sup>)</b> | <b><math>k_\tau</math><br/>(MPa<math>\cdot\mu</math>m<sup>1/2</sup>)</b> | <b>“<math>\tau_o</math>” in VPSC<br/>(MPa)</b> |
|---------------------------------------------------|----------------------------------|------------------------------------------------------------------------|--------------------------------------------------------------------------|------------------------------------------------|
| Basal Slip                                        | 9.9                              | 2.61                                                                   | 82                                                                       | 26                                             |
| Prismatic Slip                                    | 17.6                             | 5.54                                                                   | 175                                                                      | 52                                             |
| Pyramidal Slip                                    | 40*                              | 9.86*                                                                  | 311*                                                                     | 101                                            |
| Extension<br>Twin                                 | 5.7                              | 3.48                                                                   | 110                                                                      | 27                                             |

\* $\tau_o$  for pyramidal is obtained from the literature [67] and  $k_\tau$  is calculated based on the dislocation pile-up theory [173].

The only difference in the input parameters between the compression and tension cases are the latent hardening parameter  $h^{twin,slip}$ , which actually help us understand the plastic anisotropy in terms of twin-slip interactions with different twin variants and twin morphologies. This issue will be discussed in detail in Chapter 6.

#### ***4.5.2 Influence of initial texture and loading path on the active deformation mechanisms***

Table 4-3 lists the ideal Schmid factors of various deformation modes active in parent grains and also in the twinned daughter grains (when the extension twin is activated) under tension and compression with different loading orientations,  $\theta$ , assuming the hot-rolled Mg plate has the perfect  $\{0002\} < 11\bar{2}0 >$  plane texture. For the twin systems, total six variants can be selected, which provide the distinct texture evolution, which will be discussed in section 4.5.3. Considering the geometric symmetry, three different variants (V1, V2, and V3) are taken into account independently. Table 4-3 clearly illustrates that the Schmid factors for various deformation mechanisms are highly dependent on both loading path and loading orientations. Using Table 4-3 and the CRSSes of various deformation modes as a guideline, let us discuss the evolutions of the active deformation modes presented in Figure 4-4 and Figure 4-5.

For tension along  $0^\circ$ , the extension twin is the easiest mode to be activated in terms of the crystal orientation because the Schmid factor is close to the ideal value of 0.5. It is known that for hexagonal close-packed structure, when  $c/a$  ratio is close or smaller than 1.633 such as for Mg, the extension twin occurs when  $c$ -axis is extended.

Table 4-3. Ideal Schmid factors,  $m$ , calculated for basal slip, prismatic slip, pyramidal slip, and extension twin as a function of the loading orientation,  $\theta$ , between the LD and ND for uniaxial tension and compression cases. The ideal Schmid factors presented here are the maximum value for each case calculated assuming a perfect  $\{0001\} < 10\bar{1}0 >$  plane texture. The twin variants; V1, V2, and V3; represent  $(10\bar{1}2)[\bar{1}011]$ ,  $(01\bar{1}2)[0\bar{1}11]$ ,  $(\bar{1}102)[1\bar{1}01]$ , respectively

| Loading Path | $\theta$ (°) |          | Basal | Prismatic | Pyramidal | Extension Twin |       |       |
|--------------|--------------|----------|-------|-----------|-----------|----------------|-------|-------|
|              |              |          |       |           |           | V1             | V2    | V3    |
| Tension      | 0 (ND)       | Parent   | 0     | 0         | 0.446     | 0.499          | 0.499 | 0.499 |
|              |              | Daughter | 0.056 | 0.431     | 0.357     | -              | -     | -     |
|              | 22.5         | Parent   | 0.354 | 0.036     | 0.445     | 0.351          | 0.426 | 0.391 |
|              |              | Daughter | 0.343 | 0.349     | 0.337     | -              | -     | -     |
|              | 45           | Parent   | 0.500 | 0.117     | 0.280     | 0.050          | 0.258 | 0.105 |
|              |              | Daughter | 0.429 | 0.188     | 0.294     | -              | -     | -     |
|              | 67.5         | Parent   | 0.354 | 0.269     | 0.445     | -              | -     | -     |
|              | 90 (TD)      | Parent   | 0     | 0.433     | 0.446     | -              | -     | -     |
| Compression  | 0 (ND)       | Parent   | 0     | 0         | 0.446     | -              | -     | -     |
|              | 45           | Parent   | 0.500 | 0.117     | 0.280     | -              | -     | -     |
|              | 90 (TD)      | Parent   | 0     | 0.433     | 0.446     | 0.374          | 0     | 0.374 |
|              |              | Daughter | 0.056 | 0.003     | 0.468     | -              | -     | -     |

This represents two loading conditions: compression perpendicular to the  $c$ -axis or tension parallel to the  $c$ -axis [29]. For the rolled Mg plate, the extension twinning is possible to be activated, when the tensile loading orientation  $\theta_T$  is lower than  $45^\circ$ .

The dominant deformation mechanisms change with the loading path and loading orientation, which is summarized in Table 4-4. Considering that the  $\{10\bar{1}2\}$  extension twin re-orientates the  $c$ -axis about  $86.3^\circ$  and the same angle shifts shown in the pole figures (Figure 4-6, Figure 4-7, and Figure 4-8), the dramatic rotation of crystals observed in the  $0^\circ$  tension is due to the deformation twin. The crystal rotation, in turn, causes the changes in Schmid factors for subsequent deformation activities within the daughter grains.

For tension along  $0^\circ$ , with further increase in strain, the dominant deformation mechanisms change to the prismatic slip in the daughter grains, while the extension twinning is still most favorable in the parent and the basal slip remains unfavorable throughout, Table 4-3 and Table 4-4. The higher CRSS to activate prismatic slip as well as the higher hardening parameters of the prismatic slip result in the dramatic increase in the macroscopic hardening rate shown in the strain range of 8% and 15%. The Schmid factors of the  $\langle c+a \rangle$  pyramidal slip for the specimens with various initial textures are all relatively high, which means it is relatively easy to be activated in terms of the crystal orientation. However, the highest CRSS of the pyramidal slip compared to all the other deformation modes limits the  $\langle c+a \rangle$  slip to a very high applied stress levels. The simulation results reveals that the activation of  $\langle c+a \rangle$  pyramidal slip systems for all of the tension samples with different initial texture occurs at high stress level, which agrees well with the literature [30, 31].

Table 4-4. Summary of active plastic deformation mechanisms at yielding and subsequent hardening stages under uniaxial tension or compression for various loading orientation,  $\theta$ , between the LD and ND.

| Loading Path | $\theta$ (°) |          | Yield    | Flow                                  |
|--------------|--------------|----------|----------|---------------------------------------|
| Tension      | 0 (ND)       | Parent   | T        | T                                     |
|              |              | Daughter | -        | Pr $\rightarrow$ Py                   |
|              | 22.5         | Parent   | T + B    | B $\rightarrow$ Pr $\rightarrow$ B+Py |
|              |              | Daughter | -        | B $\rightarrow$ Pr $\rightarrow$ B+Py |
|              | 45           | Parent   | B (+ T)  | B                                     |
|              |              | Daughter | -        | B $\rightarrow$ Pr $\rightarrow$ B+Py |
| Compression  | 67.5         | Parent   | B (+ Pr) | B+Pr $\rightarrow$ Py                 |
|              | 90 (TD)      | Parent   | Pr (+ B) | Py                                    |
|              | 0 (ND)       | Parent   | B        | Py                                    |
|              |              | Daughter | -        | -                                     |
|              | 45           | Parent   | B        | B+Py                                  |
|              | 90 (TD)      | Parent   | Pr (+B)  | T                                     |
|              |              | Daughter | -        | B $\rightarrow$ Py                    |



For the 22.5° tension case, both basal slip and twinning have the highest Schmid factor with the similar CRSSes, and, therefore, both deformation modes are activated simultaneously at the yielding. As twinning progresses, the crystal re-orientation alters the deformation activities within the daughter grains, where both basal and prismatic slips have high Schmid factors. But the basal slip has much higher activities because of its relatively lower CRSS. Therefore, the texture hardening effect caused by the extension twin in 22.5° tension is relatively low compared to that of the 0° case.

Basal slip is the dominant deformation mode in the  $\theta_1=45^\circ$  case due to its high Schmid factor and low CRSS. Some extension twin occurs as well. In this loading orientation, the maximum Schmid factor for the twin decreased to 0.258 for all three twin variants. Therefore, the extension twin gets activated when the stress is relatively higher. Moreover, as shown in Figure 4-1b, there is a slight angular distribution of the basal pole from the ND in the initial texture. During the tension along 45°, the grain sets that are off ND towards the LD have higher Schmid factors for the extension twin than the grains that are more parallel to the ND. Thus, the extension twin is still relatively easy to be activated in the off-ND grains and results in the crystal reorientation. However, since only those set of grains oriented towards the LD is under tension along *c*-axis, the overall twin volume fraction is much lower than that observed in 0° and 22.5° cases. The basal slip is by far the most favorable mode in the daughter grains as well, which provide the relatively high activity of basal slip in the material throughout the whole plastic deformation process.

For the  $67.5^\circ$  tension case, the basal slip is activated since it has the relatively higher Schmid factor. However, the Schmid factor of basal slip is lower than that in  $45^\circ$  case so that the yield strength is higher. Some prismatic slip is also activated with the increase in the strain. The extension twin is almost impossible to be activated even for the grains that are off ND and almost all of them are under compressive loading along the  $c$ -axis.

For the  $90^\circ$  tension case, the prismatic slip is the dominant deformation mode since it has the ideal crystal orientation. Basal slip is in the non-favorable orientation so that it is very hard to be activated even though it has lower CRSS.

In the compression along  $0^\circ$  case, the extension twin cannot be activated since the  $c$ -axis is under compression in this loading path. The Schmid factors for both basal slip and prismatic slip are 0 if the crystals have the ideal orientation, which means they cannot be activated either. However, as we have seen already, the initial texture shows that the  $\{0002\}$  poles have angular distribution around ND, which means the Schmid factors for the basal and prism slips are low but not zero. Therefore, at high normal stress basal slip can be activated first with its lower CRSS than that of prismatic slip. On the other hand, the pyramidal slip has much higher Schmid factor (0.446) in this case but high CRSS as well. Thus, it gets activated when the applied stress is high. Overall, the basal and pyramidal slip are competitive in this case, but considering that only the grains with the high angular deviation from the ideal orientation are favorable for basal slip, pyramidal slip dominates most part of the plastic deformation.

Basal slip has the favorable orientation factor in the compression along 45° case, which is similar to the tension along 45° case. Therefore, the low yield strength is observed in this loading path. However, the twinning is very limited since most of the grains are under the compression along *c*-axis.

For the compression along 90° case, the prismatic slip in the matrix has the highest Schmid factor (0.433), which suggests that it should be highly active in this case. Moreover, the extension twin has the relatively high Schmid factor (0.374) as well. Considering the higher CRSS of prismatic slip than that of extension twinning, the extension twinning and prismatic slip are two competitive modes. According to the VPSC simulation, the prismatic slip occurs earlier than extension twinning and it is quickly replaced by the extension twin with the increase in strain. As the extension twin grows and the {0002} poles shift from the ND to TD (in Figure 4-8), the extension twin rotates the *c*-axis from ND towards TD by 86.3° making the *c*-axes of the daughter grains almost parallel to the TD. As a result, the Schmid factors of various deformation modes in the daughter grains changes significantly, Table 4-4. It shows that both basal and prismatic slips have relatively low Schmid factors (0.087 and 0.003) in daughter grains. Therefore, high strain hardening is observed between the strains of 0.075 and 0.125. This texture hardening comes from the crystal reorientation, which is clearly predicted by the VPSC simulation. With the increase in the applied strain, the pyramidal slip is activated in the daughter grains as well.

The evolutions and interactions of various dominant deformation modes in both parent and daughter grains discussed above are summarized in Table 4-4.

#### ***4.5.3 Effect of the complex interaction of deformation mechanisms on the texture evolution***

Based on the relationship between the relative activities of various deformation modes and texture evolutions, it is revealed that the extension twin leads to the significant texture changes during the deformation and influence the subsequent deformation mechanisms active in the twinned daughter grains. However, the texture evolution caused by the  $\{10\bar{1}2\}$  extension twinning under the tension along ND is significantly different with that under the compression along TD or RD direction, which has been reported previously in the literature. Several reports [32-34] revealed that the  $\{0002\}$  poles rotate to TD when compressed along TD, while the  $\{0002\}$  poles rotate to RD when compression along RD. Overall, for the in-plane compression case,  $\{0002\}$  poles rotate towards a certain orientation instead of spreading into various orientations in the TD-RD plane. On the other hand, in the tension along ND case, the  $\{0002\}$  poles seem randomly rotate onto an orientation within the TD-RD plane. It is also demonstrated that the entire grain reorients to the new orientation after extension twinning under the compression perpendicular to the  $c$ -axes of the crystals. In this process, a few narrow twin bands, which are parallel with each other nucleate and propagate in the parent. With the increase in the stress, the narrow twin grows until it meets another twin band within the grain, which has the same orientation and they merge as a larger twin. The twin growth typically dominates the twinning process and finally results in a rapid increase in twin volume fraction [34] in this case.

On the other hand, the extension twin activated under the tension parallel to the  $c$ -axis follows another path. All twin variants can be equally activated if the rolled plate has the ideal plane texture because of the Schmid factors for these three variants are identical. Therefore, the  $\{0002\}$  poles of daughter grains can ‘choose’ various orientations on the TD-RD plane and form a circumferential rim along TD-RD plane of the pole figure. In order to understand the twin variant selection, six twin variants including: (V1-V6) are considered here, Figure 4-9a. The difference may come from the PTR scheme used in the VPSC simulation, since only one predominant variant is allowed in the PTR scheme in each “grain” (orientation). In the PTR scheme, one variant is the most preferred and once it is selected, it becomes the dominant variant. No other variants are allowed to be selected in this particular orientation. Therefore, the rim in the  $\{0002\}$  PF still shows up at the beginning of the deformation, since the orientation input matrix is large enough in the simulation. The deviation of the initial grain orientations in the VPSC as an input allows multiple variants to be selected overall, even though only one is allowed in each orientation. However, there is no chance to make the second or third variants to be selected during the deformation when the applied stress rises, the preferred variants is overestimated compared to the real measured cases. Therefore, the intensity of V3 in the VPSC is somewhat overestimated.

The extension twin induced texture evolution is also observed at tension along  $22.5^\circ$  and  $45^\circ$  cases. For  $22.5^\circ$  case, the extension twin is still the dominant deformation mechanisms at the beginning stage of plastic deformation, which facilitate significant crystal reorientation.

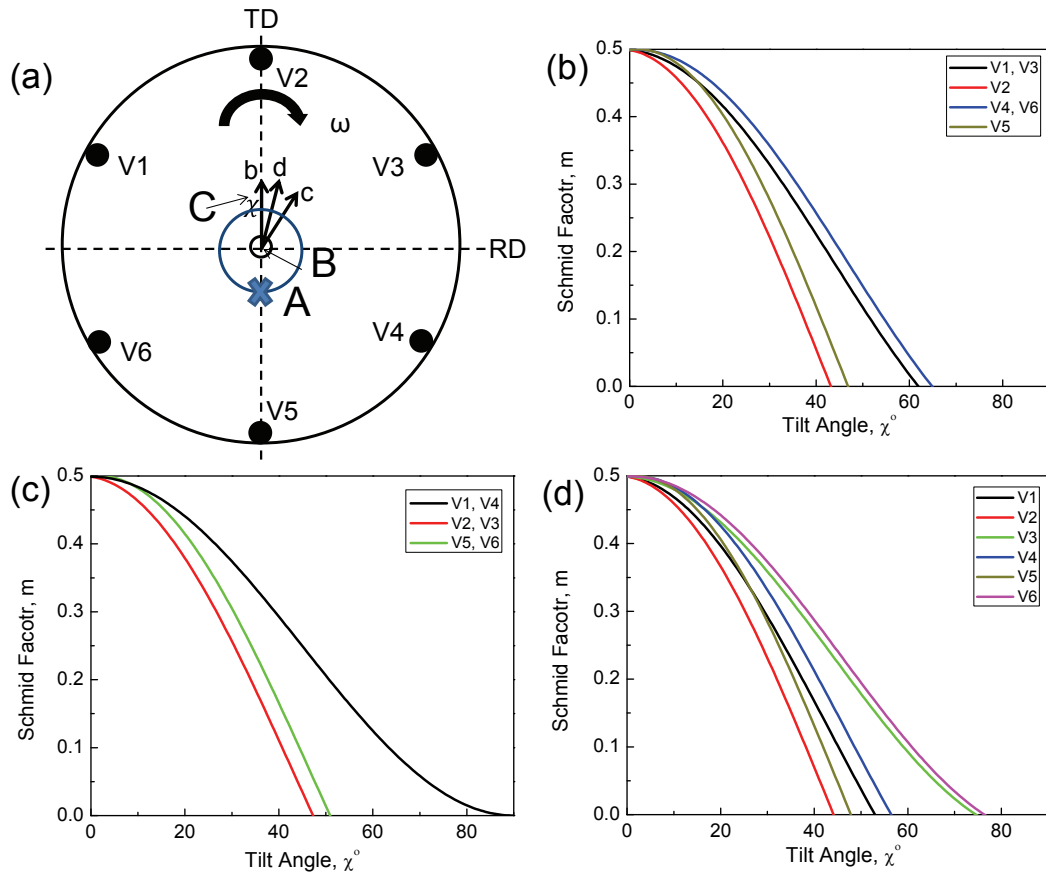


Figure 4-9. The illustration of the grain orientation deviation and the tilting of the loading orientation in the {0002} pole figures and the Schmid factor changes with the changes of the tilting angle,  $\chi$ , between the {0002} pole and loading orientation LD.  $\times$  in the Figure 4-9a represents the loading orientation. (a) illustration of the grain orientation deviation

If we assume the grain has the perfect orientation  $\{0002\} \langle 11\bar{2}0 \rangle$ , the loading orientation is tilted along TD for the angle  $\chi$  in the PF (same as  $\theta_T$ ) and the Schmid factors of six variants change with the increase in  $\chi$ . Moreover, the Schmid factors for the six variants even at a given  $\chi$  are not constant. If the  $\chi$  is along orientation b shown in Figure 4-9b, the Schmid factor for V4 and V6 is the highest one at the tilting angle  $\chi$  ranging from  $0^\circ$ - $65^\circ$ . Similarly, if the loading angle tilts in the opposite direction, the V1 and V3 possess the highest Schmid factor, while the Schmid factor of V2 is slightly lower. Therefore, for the grains with the  $\{0002\}$  along ND (a cross marked as A in Figure 4-9a), the angle between the LD and  $\{0002\}$  pole is  $\chi$  and V1 and V3 have the highest Schmid factor. For the grains at B, all of 6 variants have the same Schmid factor and the homogenous twin band forms. For the grains at C, the  $\chi$  is very high so that the twin is very hard to be activated since the Schmid factor decreases with the increase in  $\chi$ . Therefore, a twin band can be formed after the activation of extension twin with the preferred orientations at V1 and V3 in the  $\{0002\}$  PF. This is observed in the texture evolution obtained through the VPSC simulation, Figure 4-7. The twin band forms at the top of the  $\{0002\}$  PF, which is observed in the texture evolution measurement through S-XRD. However, the twin preferred orientation selects V2 instead. The difference shown in the VPSC simulation may come from the stress concentration and the approximation of HEM scheme used in the VPSC simulation, which is illustrated in [27].

In  $45^\circ$  case, the basal slip is the dominant deformation mode instead of extension twinning. However, some grains are still favorable for twinning as we discussed in the previous section. For the grains with the  $c$ -axes deviated from the ideal orientation along

A direction (Figure 4-9a), the angle between the loading orientation and  $c$ -axis of the grains are actually smaller than  $45^\circ$ , which facilitates higher Schmid factors than the ideal parent orientation and twinning may be activated in those grains. As a result, the intensity of this part decreases by shifting the  $\{0002\}$  pole to the rim at the north of the PF, which is similar to the case of  $22.5^\circ$ . However, the twin has less activation or even cannot be activated in the grains which have the deviation of basal component in the opposite direction (along C region), since the angle between the loading orientation and  $c$ -axis of these grains are not ideal for the extension twin. Thus, the component in the center of  $\{0002\}$  PF depletes asymmetrically.

For the texture evolution of  $67.5^\circ$  and  $90^\circ$  cases, which are dominated by basal slip and/or prismatic slip, the texture features remains unchanged during the deformation, which indicates that  $\langle a \rangle$  type dislocation does not provide obvious texture evolution during the deformation at room temperature under the given amount of strain. However, the slight change of basal component for all of five tension cases towards the end of deformation, when  $\langle c+a \rangle$  pyramidal slip is activated, indicates that  $\langle c+a \rangle$  pyramidal slip may tilt the basal component towards to the TD during the deformation, Figure 4-5 and Figure 4-6.

#### ***4.5.4 Twin volume fraction evolutions: comparison between S-XRD and VPSC data***

Considering the texture evolutions caused by the extension twin as discussed earlier, the twin volume fraction can be estimated through the integration of the intensity of the basal poles re-oriented to (or from) a particular angle. For the parent, the basal



poles are initially almost parallel to the ND, which is shown by the initial texture shown in Figure 4-1b. The basal poles rotate about  $86^\circ$  from the ND, when the extension twin occurs. Therefore, the intensity is zero at the orientation towards the ND between  $40^\circ$  and  $90^\circ$ , and thus, one can use the ratio of the integrated intensity of the area where the angle towards the ND between  $45^\circ$  to  $90^\circ$  and that between  $0^\circ$  and  $45^\circ$  as a direct measure of the twin volume fraction. Note that the selection of  $45^\circ$  to be the separating point for the determination of twin volume fraction is reasonable for the current case, while it is not necessarily appropriate for other texture cases. The twin volume fraction evolution as a function of applied strain for different tension LDs is shown in Figure 4-10. The open symbols represent the twin volume fraction obtained from the integration of the intensity of the PFs measured by S-XRD and the solid lines are the data from VPSC simulation.

The VPSC simulation and S-XRD measurement agrees well for all cases studied and the results demonstrate that the twin volume fraction decreases with the increase in the  $\theta_T$  for the tension cases, Figure 4-10 (a-b). The twin volume fraction increases with the increase in the strain. For tension along  $67.5^\circ$  and  $90^\circ$  cases, the volume fractions of the extension twins are very small. In general, the activation of extension twin is slower in the measured S-XRD data than that of the VPSC simulation. The maximum twin volume fraction of extension twin obtained through the S-XRD is about 60% of that obtained through VPSC simulation for the tension along  $0^\circ$  and  $22.5^\circ$  cases.

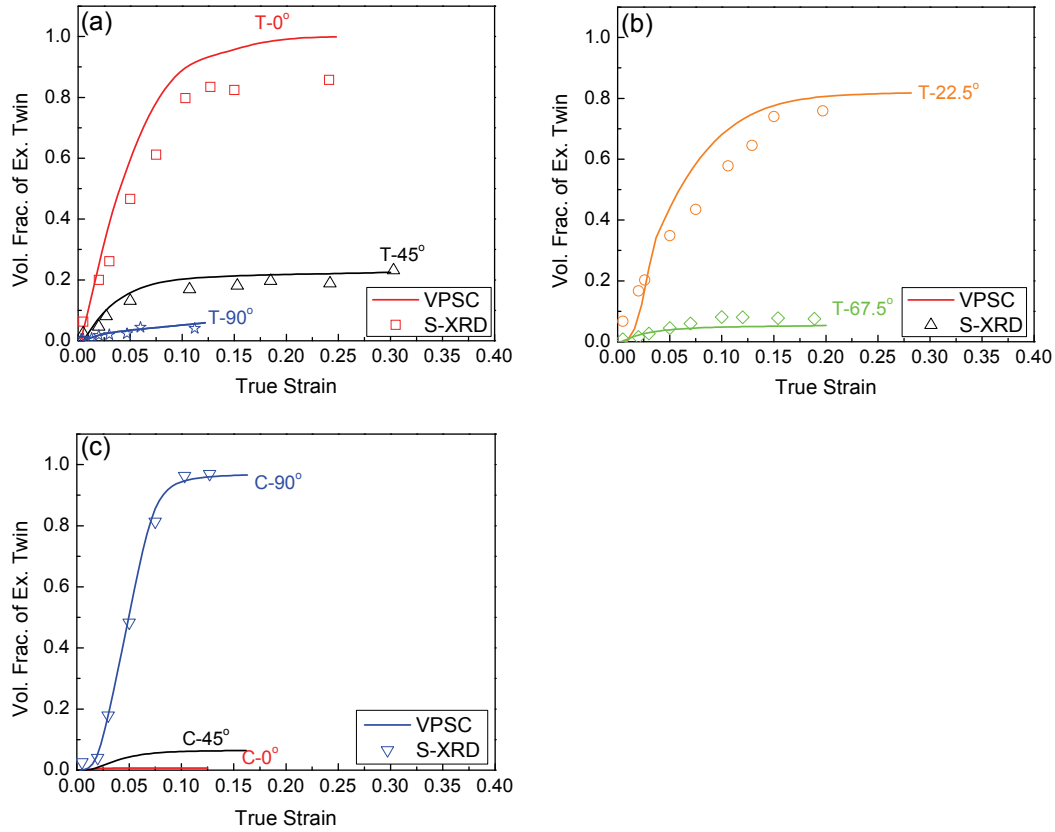


Figure 4-10. Evolution of volume fraction of extension twin evolution as a function of strain under uniaxial tension and compression with different loading orientation,  $\theta$ , respect to the ND of the rolled plate. The symbol represents the data obtained by the integration of  $\{0002\}$  pole intensity of the area where the angle towards to the ND is between  $45^\circ$ - $90^\circ$  based on the PFs measured through S-XRD and the solid lines represents the data obtained through the VPSC simulation, (a) uniaxial tension at  $0^\circ$  and  $22.5^\circ$ , (b) uniaxial tension at  $45^\circ$ ,  $67.5^\circ$ , and  $90^\circ$ , and (c) uniaxial compression.

The difference between the volume fraction obtained by S-XRD and VPSC may come from the PTR scheme used in the VPSC simulation as well as the texture insensitivity for the narrow twin bands.

The twin volume fraction as a function of strain is examined by S-XRD for the compression along 90° case as well to compare with the VPSC simulation, Figure 4-10c. Overall, the experimental result matches the VPSC simulation in this case very nicely. It was found that the twin volume fraction increases rapidly in the strain range from 0.025 to 0.08. When the strain is at 0.08, the twin volume fraction is almost up to 1.0, which means almost all the matrix reoriented due to the twinning. The twin volume fraction increases with the increase in the  $\theta_C$ , which is opposite with the tension case.

#### ***4.5.5 Hardening rate and active deformation modes***

The work hardening rates  $h$  ( $= d\sigma/d\varepsilon$ ) as a function of flow strain ( $\sigma - \sigma_o$ ) for cases with different LDs can be calculated from the stress-strain behaviors obtained from the mechanical test (solid lines) and VPSC modeling (open symbols), Figure 4-11. For the sigmoidal type hardening behaviors observed in extension twin dominant cases (i.e.,  $\theta_T = 0^\circ$  and  $\theta_T = 22.5^\circ$ ), the hardening rate  $h$  shows a concave-up parabolic profile. The hardening rate increases with the strain until the stress is up to 150MPa and 130MPa, respectively for the two cases and arrive the maximum point. With the continuous increase in the stress, the hardening rate decreases.

For the slip dominant deformation cases ( $\theta_T = 45^\circ$ ,  $67.5^\circ$ , and  $90^\circ$ ), the initial rapid linear drop in work hardening rate indicates the elasto-plastic (slip) transitions.

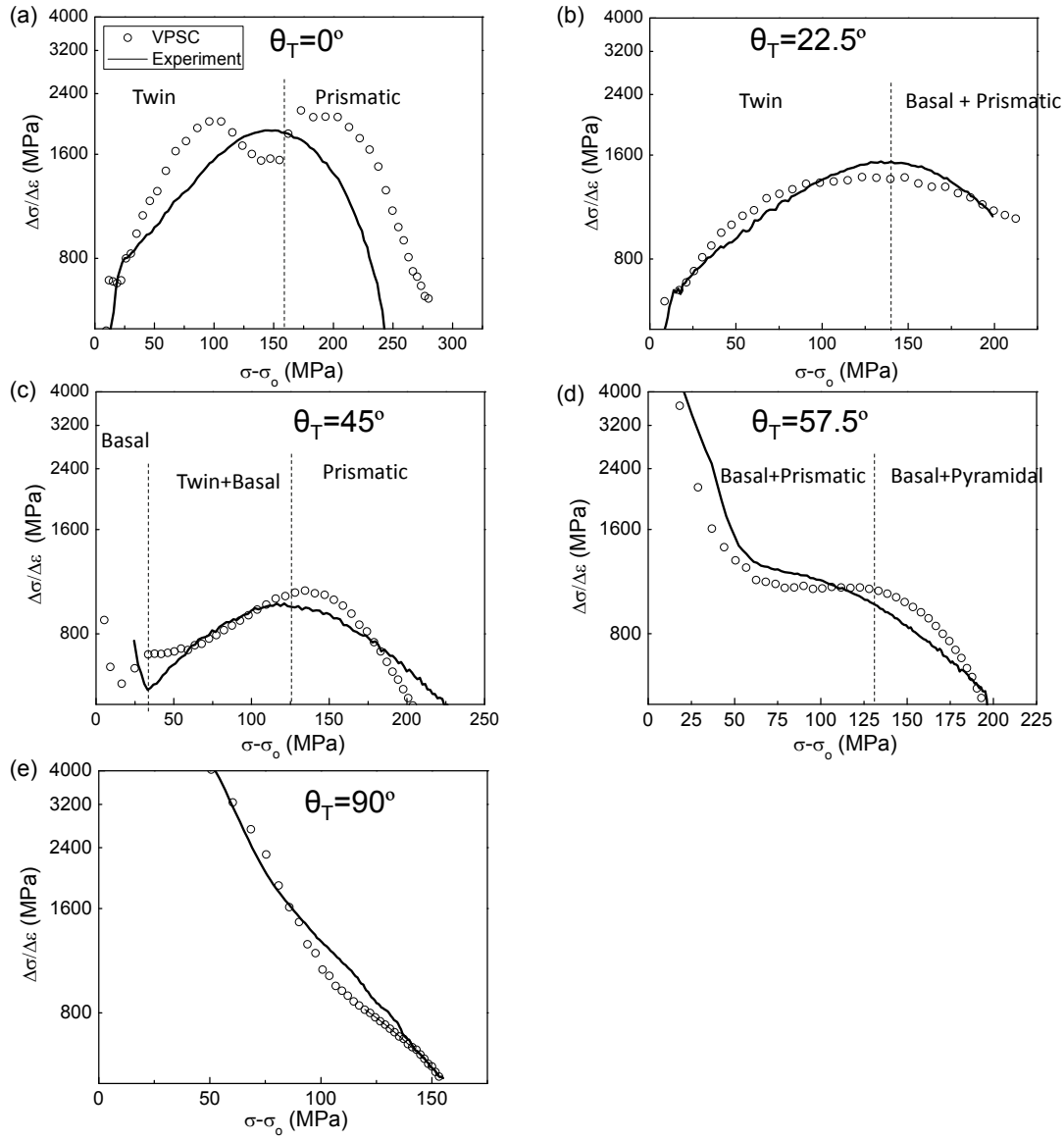


Figure 4-11. Hardening rate  $\Delta\sigma/\Delta\epsilon$  as a function of flow stress obtained from the experimental stress-strain curves (open symbol) and VPSC simulated curves (solid lines) under the tension along different loading orientation respects to normal direction (ND) of the Mg plate,  $\theta_T$ , (as shown in Figure 4-1c) simulated through VPSC modeling. (a)  $\theta_T=0^\circ$ , (b)  $22.5^\circ$ , (c)  $45^\circ$ , (d)  $67.5^\circ$ , and (e)  $\theta=90^\circ$ .

In this region, some grains with slip-favorable orientations have already yielded, while others with harder orientations are still in the elastic deformation stage. With the increase in the stress, more and more grains transfer to the plastic deformation. After the elasto-plastic transitions, the steady work hardening stages with different work hardening rates follows, which is the stage III hardening range. The normal work hardening rates should gradually decrease with the increase of stress in the stage III. However, only the hardening behavior for 67.5° and 90° tension cases show the normal hardening trends. For the tension  $\theta=67.5^\circ$  case, the hardening rates keep decrease with the increase in stress. However, the decrease can be separated into two stages. Stage III-1 is the flow stress,  $(\sigma - \sigma_o)$ , range from 55 to 100MPa and Stage III-2 ranges from 100 to 200MPa. These two stages have two different slopes. For  $\theta_T=45^\circ$  case, the Stage III is even more complicated. It shows an ‘abnormal’ increase stage at the stress lower than 125MPa and followed by a ‘normal’ decrease curve.

According to Lukac’s theory [35], the work hardening is the combination results of the propagation of dislocations and the annihilation of dislocations. The work hardening rate  $h$  can be expressed as:

$$\frac{d\sigma}{d\varepsilon} = h = \frac{A}{\sigma - \sigma_{0.2}} + B - C(\sigma - \sigma_{0.2}) - D(\sigma - \sigma_{0.2})^3 \quad 4-10$$

where

$$A = \frac{(\alpha G)^2}{2} \cdot \frac{b}{S} \quad 4-11$$

$$B = \frac{M\alpha G}{k_1} \quad 4-12$$

and

$$C = \frac{M\alpha Gbk_2}{2} \quad 4-13$$

The first term in equation 4-10 is related to the interaction of dislocations with the non-dislocation obstacles such as the solute content and precipitation. The second term represents the interaction of dislocation with the forest dislocation and the third terms is related to the dislocation cross-slip, while the forth term is related to the climb of the dislocations. The parameter A can be simplified as 0 considering the low solute content and few precipitations in the AZ31 Mg alloy. Meanwhile, the forth term, which is related to the dislocation climb can also be ignored when the materials is deformation at room temperature. Therefore, the equation 4-10 can be simplified as:

$$\frac{d\sigma}{d\varepsilon} = h = B - C(\sigma - \sigma_{0.2}) \quad 4-14$$

In equation 4-11- 4-13, M is the orientation factor,  $\alpha$  is the constant related to the dislocation interaction, G is the shear modulus and b is the Burgers vector of the dislocation. S represents the grain size.  $k_1$  is the geometrical factor and  $k_2$  is the coefficient of the dislocation annihilation intensity. Therefore, it is clear that the work hardening rate in stage III is related to the crystal orientations, Burgers vectors of dislocations, and ability of cross-slip for various defamation modes. Therefore, the hardening behavior changes with the dominant deformation modes and the crystal orientation factor, M, which is highly dependent on the activation of extension twin, which introduce the significant texture evolution as we discussed earlier.

In order to understand the changes in hardening behavior with the changes in the deformation modes, the flow of the dominant deformation mode is noted in Figure 4-11 and Figure 4-12 based on the relative activities obtained from the VPSC simulation.

In the tension along  $0^\circ$  and  $22.5^\circ$  cases, the parabolic hardening behavior shows with the increase in the strain. At the beginning of the yielding, the hardening rate is very low, since the extension twin, which does not have any self-hardening effect, is the dominant deformation mode. The hardening rates keep increasing with the increase in strains until the strain up to 0.145 and reach maximum. In this stage, the activation of prismatic slip increases with the increase in the strain inside the twinned daughters. Considering the much higher CRSS of prismatic slip than that of the extension twinning, higher stress is required to activate the prismatic slip to accommodate the strain. Moreover, prismatic slip has a much higher self-hardening coefficient compared to the extension twin because of the dislocation-dislocation interaction and dislocation-boundary interaction. Therefore, with the increase in the relative activation of prismatic slip in the daughter grains, the hardening rate continue to increase. After the strain reaches 0.145, the extension twinning has almost finished and the plastic deformation changes to pure slip dominant mode for both of these tension cases.

In tension along  $45^\circ$  case, the hardening rate can be describe as a sharp decrease followed by a slightly increase and then by a decrease. When the strain is lower than 0.175, the hardening rate increases with the increase of strain.

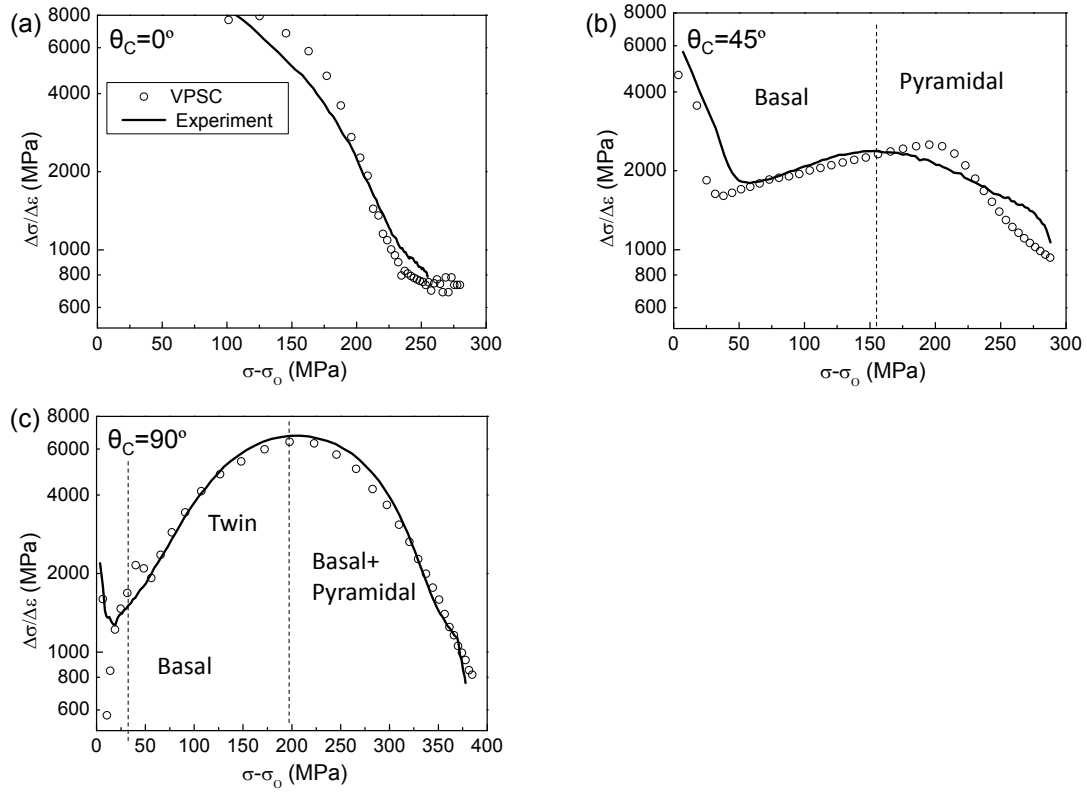


Figure 4-12. Hardening rate  $\Delta\sigma/\Delta\epsilon$  as a function of flow stress obtained from the experimental stress-strain curves (open symbol) and VPSC simulated curves (solid lines) under the compression along different loading orientation respects to normal direction (ND) of the Mg plate,  $\theta_C$ , (as shown in Figure 4-1c) simulated through VPSC modeling. (a)  $\theta_C=0^\circ$ , (b)  $45^\circ$ , and (c)  $90^\circ$ .



In this stage, basal slip is the dominant deformation mode, while the relative activity of prismatic slip gradually increases and replaces some part of the shear is accommodated by basal slip with the increase in strain. According to the equations 4-12 and 4-14, the hardening rate will increase with the increase of orientation factor  $M$ , considering the same Burgers vector for basal and prismatic  $\langle a \rangle$  dislocations. The prismatic slip has relatively higher orientation factor than basal slip because of the lower Schmid factor as listed in Table 4-3. Moreover, the extension twin influences on the hardening behavior as well due to the texture evolution, which makes the orientation factor of basal slip increase in the daughter grains even though the total volume fraction of extension twin is limited in this case. Therefore, the hardening rate increases with the increase of the strain because of the increase of the orientation factor caused by the extension twinning and activation of prismatic slip, which is indicated by the decrease of Schmid factors as listed in Table 4-3.

On the other hand, the texture almost stays the same during the deformation in the tension along  $67.5^\circ$  and  $90^\circ$  cases so the changes in the hardening behaviors are the result of the changes in the dominant deformation modes with the increase in strain. In  $67.5^\circ$  case, the Stage III is separated into two different parts at about 0.10 strain. The first part has a much lower dropping rate compared to the second part. It is clearly shown that the pyramidal slip becomes active when the strain is about 0.10 in Figure 4-11d. Therefore, it indicates that the pyramidal slip has much higher rate of dislocation annihilation compared to the basal or prismatic slip, which dominated the deformation when strain is lower than 0.10. It has been reported that  $\langle c+a \rangle$  pyramidal slip is easier to cross slip since there are more slip planes available, which explains the higher  $C$  value in equation

4-14. In the 90° case, the pyramidal slip is activated at the beginning of the plastic deformation and gradually replaces prismatic slip with the increase of strain and stress. Thus, Stage III in this case remains rather simple.

For the compression cases, the hardening rate decreases with the increase of the flow stress when  $\theta_c=0^\circ$ , since only slip systems are activated in this loading path, Figure 4-12a. The hardening rate as a function of net flow stress is shown in Figure 4-12b for the compression along 45° case. The hardening rate decreases with the increase in the flow stress when the net flow stress is lower than 50MPa (strain at 0.04) followed by the moderate increase in the flow stress range of 50-150MPa (strain as 0.04 to 0.06). This is related to the activation of extension twinning. The hardening rate decreases again when the flow stress is higher than 150MPa. In the compression along 90° case (Figure 4-12c), the hardening rate shows the parabolic profile. It increases dramatically to 6200MPa when the strain ranges from 0.02 to 0.08. In this stage, the volume fraction of extension twin increase rapidly until it reaches close to 100%. The activation of twinning reoriented the grains which enables basal and prismatic slips in the relatively hard orientations. Therefore, the dramatic increase in the orientation factor,  $M$ , increases the hardening rate in this stage. After the crystal is reoriented, the slip systems become the dominant deformation mode, which causes the normal decrease in the hardening rate with the increase in the stress. It is also worth noting that when the strain is lower than 0.02 (net flow stress lower than 25MPa), the hardening rate decreases with the increase in the flow stress due to the dominant prismatic slip here, which is activated earlier than the extension twin. It was also found that the hardening behavior during compression along

90° has some similar aspects with that under tension along 0° case such as the parabolic profile, since both of them are the twin active paths. However, the peak hardening rate of the 90° compression is much higher than that of 0° tension. Moreover, at the beginning stage, a normal decrease trend shown in the 90° compression is not found in the 0° case. The difference in the hardening behaviors for these two cases will be discussed in detail in Chapter 6 in terms of the twin-twin, twin-slip interaction, and texture hardening.

#### 4.6 Conclusions

A commercial hot-rolled Mg AZ31B alloy with various initial textures was studied under uniaxial tension and compression at room temperature using synchrotron x-ray diffraction and visco-plastic self-consistent (VPSC) modeling to understand the interplay between deformation mechanism and texture on the plasticity of Mg alloy. The conclusions are as follows.

1. One set of input crystal plasticity parameters, including  $h_0^\alpha$ ,  $h_1^\alpha$ ,  $\tau_0^\alpha + \tau_1^\alpha$  for the deformation mechanisms of  $\{0001\} \langle 11\bar{2}0 \rangle$  basal slip;  $\{\bar{1}010\} \langle 11\bar{2}0 \rangle$  prismatic slip;  $\{10\bar{1}1\} \langle 11\bar{2}3 \rangle$  pyramidal slip, and  $\{10\bar{1}2\} \langle \bar{1}011 \rangle$  extension twin was used in the VPSC model to achieve good agreements with the measured stress-strain behaviors and texture evolutions for both the tension and compression at various loading orientations. The  $\tau_0^\alpha$  for various deformation modes are obtained through the Hall-Petch relationship study for each deformation mode. Only the latent hardening parameter  $h^{twin,slip}$  was changed between the tension and compression cases, which provide insight to the twin-slip interactions introduced by different twin morphologies.

2. Various deformation mechanisms dominate the plastic deformation for the specimens with different initial textures. The extension twin is activated first during the plastic deformation at the tension along  $0^\circ$  and  $22.5^\circ$  cases. The prismatic and basal slips are the dominant deformation mechanisms at the twinned daughter grains in these two cases, respectively. The basal slip is activated first at the tension along  $45^\circ$  and  $67.5^\circ$  cases. However, some extension twin is still activated at the tension along  $45^\circ$  since the deviation of the grain orientation from the ND in the initial texture. Prismatic slip dominates the plastic deformation at the tension along  $90^\circ$  case followed by a pyramidal slip. The twin volume fraction decreases with the increase in the loading angle during the uniaxial tension.

3. The basal slip is activated first with very limited activities and followed by the pyramidal slip in the compression along  $0^\circ$  case. The basal slip is the dominant deformation mode at the  $45^\circ$  case. In the compression along  $90^\circ$ , the prismatic slip is activated first at the yielding stage and followed by the extension twin. Basal slip is the dominant deformation mode followed by the pyramidal slip in the twinned daughter grains in the compression  $90^\circ$  case.

4. The extension twin results in the most significant texture evolution, which makes the  $c$ -axis of the crystal shift about  $84^\circ$  during the deformation, which is observed in tension along  $0^\circ$ ,  $22.5^\circ$  and  $45^\circ$  cases. The texture evolution is dependent on the twin variants selection. The homogenous rim shows in the  $\{0002\}$  PF during the plastic deformation in the tension along  $0^\circ$  case, since multiple variants are selected based on the Schmid factor calculation, while stronger twin variant preference are observed in the

tension along  $22.5^\circ$  and  $45^\circ$  and cases. A small difference in the texture evolution was noticed between the VPSC and the S-XRD results, which is attributed to the predominant twin reorientation approximation and the self-consistent scheme used in the VPSC.

5. The hardening behaviors are analyzed for the cases with different initial textures and dominant deformation modes. It was found that the hardening rates are dependent on the dislocation characteristics and crystal orientation. The harder crystal orientation provides the higher hardening rate. The pyramidal slip has the highest dislocation recovery rate because of the easiest cross-slip, which causes rapid decrease in hardening rate with the increase in the flow stress.

## Chapter 5

### **Visco-Plastic Self-Consistent Modeling of the Hall-Petch Relationships for Various Deformation Mechanisms in a Mg Alloy**

A commercial hot-rolled AZ31B Mg alloy plate with a strong  $\{0001\} \langle 10\bar{1}0 \rangle$  plane texture was used and the texture was controlled by changing the angle  $\theta$ , between the uniaxial tensile loading direction (LD) and the normal direction (ND) of the plate to induce the activation of various combinations of plastic deformation mechanisms. The influence of the grain size on the tensile flow stress of the specimens with different initial texture was studied in terms of the effect on the activation of various deformation modes using the visco-plastic self-consistent (VPSC) simulation that incorporates Hall-Petch (H-P) relationship. The simulation was performed iteratively by fitting the stress-strain behaviors and texture evolutions to the experimental results from the specimens with different LDs and grain sizes. First, the activities of various deformation modes as a function of grain size and LD are represented. Then, the grain size effect on the flow stresses is discussed. Finally, the input parameters used for the current VPSC simulation that incorporates grain size sensitivities are discussed. The current model can be used to simulate the combined effects of texture and grain size on the mechanical behaviors.

## 5.1 Introduction

Magnesium alloys have attracted significant interests in the application of the automobile component as a light weight structure alloy. However, the poor ductility and significant anisotropy during the deformation due to the limited deformation mechanisms at room temperature are key drawbacks, which inhibit the broader application of the wrought Mg alloys. In particular, critical advances in the understanding of the combined influences of grain size and texture on the mechanical properties would enhance the applications of wrought Mg alloys. It was demonstrated in [1] that various deformation mechanisms can be obtained with the changes in the initial textures of pure magnesium or Mg alloys and, therefore, the materials show distinct plastic deformation behavior and texture developments. For magnesium and magnesium alloys [2-14] various deformation mechanisms were extensively investigated and most studies considered texture to some extent for the discussion of plastic deformation anisotropy. Moreover, simulation of mechanical properties and texture in hexagonal metals taking different slip systems and twinning systems into account has attracted increasing attention [15-20]. The effects of texture on the deformation behaviors, i.e., changing the activation of different deformation modes for polycrystal, have been studied by self-consistent model, which was demonstrated as a more effective tool to simulate the low symmetry hcp structure compared to Sachs or Taylor model especially for the anisotropic behaviors. Visco-plastic self-consistent (VPSC) model is a method commonly used to simulate the large strain behavior, especially the hardening behavior and texture evolutions of Mg. Tome and Agnew [21, 22] reported that the deformation mechanisms and mechanical behaviors

are different for through-thickness compression, in-plane tension, and in-plane compression of a hot-rolled AZ31B Mg plate, which has a strong basal texture. Yi [5] simulated the deformation mechanisms and texture evolution during the compression and tension along 0, 45, and 90 degrees to the extrusion direction for hot extrusion AZ31 alloy. It was found that prismatic slip and extension twin played different roles as the loading paths and directions changed. Jain [23] reported a VPSC modeling of Mg on compression deformation behavior as a function of temperature from room temperature to 250°C for a rolled Mg plate. Four different deformation modes are considered in the modeling including basal slip, extension twin, prismatic slip, and  $\langle c+a \rangle$  pyramidal slip. It was found that the input parameters, such as  $\tau_0^\alpha$ , called critical resolved shear stress (CRSS) for various deformation modes, shows a large discrepancies in achieving good predictions of stress-strain behaviors and texture evolution. This is summarized in Hutchinson's report [24]. It was also noticed that the ratio of the  $\tau_0^\alpha$  for various deformation mode is more critical. However, the CRSS ratios also change in different polycrystal modeling approaches. For instance, the non-basal to basal slip ratio are reported to be 2:1 ~ 5:1 at room temperature. Actually, it is realized that the "CRSS" in the VPSC modeling is the resolved shear stress at a given grain size instead of the real CRSS so that it changes with the experiment conditions or the grain size of the material under investigation. Therefore, the real CRSS combined with the grain size sensitivity for each deformation mode would decrease the manually tuning processes of the VPSC model. On the other hand, Proust [25] proposed a meso-scale composite grain (CG) model, which incorporates microstructure information of twinning into the polycrystal



model to replace the predominant (PTR) scheme. The CG model can describe the twinning behaviors under the complex loading condition with both the twinning and detwinning responses. The CG model represents an ellipsoidal inclusion formed by alternating layers of twin and matrix domains. The alternating twin-matrix interfaces provide a barrier to dislocations and define a directional mean free path changes for the propagation of twinning, which, in turn, provide the hardening effect caused by the microstructure changes due to the twinning. Therefore, Hall-Petch parameters of various deformation modes need to be defined in the CG model to simulate the Hall-Petch like hardening behaviors caused by the mean free path changes of the dislocations within the twin band as described in the following equation:

$$\tau_{HP}^s = \frac{H_{HP}^s}{\sqrt{d_{mfp}^s}} \quad 5-1$$

Therefore, the grain size effect study in Mg alloys can provide a way to predict the combined effects of texture and grain size on the yielding and hardening behaviors in Mg alloy. It will also improve the understanding of the effect of twinning on the anisotropic hardening behaviors through the Hall-Petch like hardening effect.

The grain size effects have been presented earlier. The Hall-Petch (H-P) parameters including  $\sigma_0$  and  $k_y$ , have a very wide range of values in the literatures, which was summarized in Chapter 3. The H-P parameters vary when materials processing or test condition are different. For instance, the processing methods such as rolling, equal channel angular pressing (ECAP), and friction stir processing (FSP) change the H-P parameters. Moreover, the H-P parameters depend on the processing parameters

as well. 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 [26]. The H-P parameters are particularly sensitive to the loading path, which has been illustrated by comparing tension [27] and compression [28] along the extrusion direction (ED). The strength coefficient  $k_y$  (MPa·mm<sup>1/2</sup>) varies from 5.0 to 12, while the friction stress  $\sigma_o$  (MPa) ranges from 10 to 131. Therefore, the wide range of H-P parameters of Mg alloys with various initial textures under different loading paths can be expected and explained. According to dislocation pile up theory [29], which is commonly used to explain the H-P relationships in Mg alloys, the grain size sensitivity,  $k_y$ , as well as the dislocation friction stress  $\sigma_o$ , are related to the dislocation characteristics, and crystal orientation. The dislocation characteristics, such as the Burgers vector are different for each deformation mode, and the dominant deformation modes change with the changes in the crystal orientation factors. However, there are very limited reports on the H-P parameters of different deformation modes in the Mg alloys, which is the main motivation of this study.

Our previous study demonstrated systematically that various deformation mechanisms including: (i)  $\{0001\} \langle 11\bar{2}0 \rangle$  basal slip; (ii)  $\{10\bar{1}0\} \langle 11\bar{2}0 \rangle$  prismatic slip; (iii)  $\{11\bar{2}2\} \langle 11\bar{2}\bar{3} \rangle$  pyramidal slip; and (iv)  $\{10\bar{1}2\} \langle 10\bar{1}1 \rangle$  extension twinning, became dominant with the change in the tension orientation to the normal direction (ND) of rolled Mg plate. In order to understand the effect of texture on the yielding and hardening behavior of Mg AZ31B alloys, the systematical change in initial textures were obtained through change of the loading orientation towards the normal

direction (ND) of an as-received hot-rolled Mg plate (Figure 3-1), which has a strong plane texture, Figure 3-2b. By comparing the measured texture evolutions and mechanical behaviors with different loading orientations, which produce different initial textures, it was demonstrated that they can be simulated precisely through VPSC modeling with the appropriate set of input crystal hardening parameters of various deformation modes listed in Table 5-1.

The relative activities of various deformation modes under different loading orientations indicated that the different deformation modes dominate the deformation with the change of texture. Moreover, the dominant deformation modes were shifted as a function of strain because of the texture evolution during the deformation and increase in the stress due to the hardening. Each deformation system has the distinct hardening behaviors.

The grain size effect on the yielding behaviors of the AZ31B alloys with different initial texture was systematically revealed based on the Hall-Petch relationship study through the experiments. The equiaxed grains with the sizes from 26 $\mu\text{m}$  to 85 $\mu\text{m}$  (shown in Figure 3-3) were generated through the 96-hour isochronal annealing tests at various temperatures up to 723K, maintaining the texture the same as the as-received samples. In order to understand the H-P relationship for different deformation modes, various textures were generated through the different loading orientations ( $\theta=0, 22.5, 45, 67.5$ , and  $90^\circ$ ) towards the ND of the hot-rolled Mg plate as described above.

Table 5-1. Constitutive parameters of basal, prismatic, and pyramidal slips as well as extension twin for visco-plastic self-consistent modeling (VPSC) without the Hall-Petch relationship, which can fit the stress-strain behaviors and texture evolutions of the specimens with the grains size of 26 $\mu$ m.

| <b>Deformation modes<br/>(MPa)</b> | <b><math>\tau_0</math></b> | <b><math>\tau_1</math></b> | <b><math>h_0</math></b> | <b><math>h_1</math></b> | <b><math>h^*B</math></b> | <b><math>h^*P</math></b> | <b><math>h^*P</math></b> | <b><math>h^*T</math></b> |
|------------------------------------|----------------------------|----------------------------|-------------------------|-------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| Basal <a>                          | 24                         | 4                          | 2550                    | 70                      | 1                        | 1                        | 1                        | 1                        |
| Prismatic <a>                      | 52                         | 27                         | 2700                    | 300                     | 1                        | 1                        | 1                        | 1                        |
| Pyramidal <c+a>                    | 90                         | 80                         | 4000                    | 62                      | 1                        | 1                        | 1                        | 1                        |
| Extension Twin                     | 29                         | 0                          | 0                       | 0                       | 1.2                      | 1.4                      | 1                        | 1                        |

The H-P relationships for each loading orientation, and therefore initial texture, are illustrated in Figure 3-8, which shows the 0.002 offset yield strength changes as a function of  $D^{-1/2}$ , where  $D$  is the grain size. Based on the H-P study and crystal orientation factors, the H-P parameters including basal slip, prismatic slip, and extension twinning were revealed and summarized in

Table 5-2. However, the grain size sensitivity,  $k_y$ , of pyramidal slip could not be directly obtained from the experiment, since it would not be a dominant deformation mechanism at yielding no matter how texture was changed. The grain size sensitivities of basal, prismatic, and pyramidal slip were also calculated based on the dislocation pile-up theory,

Table 5-2. The calculated values of  $k_y$  for basal and prismatic slip showed good agreements with the experimental data obtained through the H-P study, which indicated that the grain boundary strengthening effect of Mg alloys could be explained through the dislocation pile-up theory.

Even though the earlier mentioned H-P study has combined the effects of texture and grain size on the tensile behaviors together, some critical issues still cannot be solved. First of all, this H-P study can only analyze the combination effects on the yielding behavior. It is not easy to understand the combination effect of grain size and texture on the flow stress or the hardening behavior directly. Jain [16] studied the grain size effect on the flow stress (2% offset strain) for the tension along transverse direction (TD) and rolling direction (RD) and found that the H-P parameters of the flow stress at different strains were different.

Table 5-2. Hall-Petch parameters of various deformation modes for AZ31B Mg alloy obtained from the experiment and also calculated based on the dislocation pile-up theory.

| Def. Mode      | $\tau_o$<br>(MPa) | $k_\tau$ (MPa $\cdot$ mm <sup>1/2</sup> ) | Calculated $k_\tau$<br>(MPa $\cdot$ mm <sup>1/2</sup> ) | Calculated $k_\tau$<br>(MPa $\cdot$ $\mu$ m <sup>1/2</sup> ) |
|----------------|-------------------|-------------------------------------------|---------------------------------------------------------|--------------------------------------------------------------|
| Basal Slip     | 9.9               | 2.61                                      | 2.89                                                    | 91                                                           |
| Prismatic Slip | 17.6              | 5.54                                      | 4.88                                                    | 154                                                          |
| Pyramidal Slip | -                 | -                                         | 9.86                                                    | 311                                                          |
| Ex. Twin       | 5.7               | 3.48                                      | -                                                       | 110                                                          |

According to our previous VPSC results, the deformation mechanisms might change as the strain increases. The grain size sensitivity is highly dependent on the deformation modes. Thus, the grain size effect on the flow stress should be different with the change of activation of deformation modes as a function of strain, which has not been systematically studied yet. Moreover, it is not easy to obtain the direct experimental value from the H-P study, since the activation of pyramidal slip was limited as a result of its much higher critical resolved shear stress than other possible deformation modes at room temperature.

Therefore, the aim of this study is to systematically analyze the combination effect of grain size and texture on the flow stress of Mg alloy through the VPSC modeling which incorporate the H-P relationship of various deformation modes.

In this chapter, we present: (1) Tensile behaviors of samples with different grain size and texture simulated through VPSC modeling; (2) the H-P relationships of various deformation modes including the prismatic slip, basal slip, pyramidal slip, and extension twinning revealed through the VPSC modeling. Most importantly, we discuss the grain size effect on the flow stress and hardening behavior. The better understanding of the H-P parameters of different deformation modes in Mg can be used to improve the polycrystal modeling of Mg alloy with various grains size and texture. It is important to be able to predict the mechanical properties of Mg alloy after thermo-mechanical processing, which often changes the grain size and texture simultaneously and significantly.

## 5.2 Experimental and Simulation

### 5.2.1 *Materials and experiments*

The material used in the study is a commercial AZ31B Mg alloy which was hot rolled with O-temper annealing condition. The dimension of plate was 400×250×90mm, which provided adequate dimensions for a tension test at various angles between normal and transverse directions of the plate. Throughout the chapter, ND, TD, RD, and LD refer to normal, transverse, rolling, and tensile loading direction, respectively. Four grain size cases including 25μm as the as-received condition, 43μm, 65μm, and 83μm, were obtained through isochronal annealing for 96 hour at different temperatures ranging from 623K to 803K, as shown in Figure 3-3. The texture of specimens with various grain sizes after annealing were the same as that of the as-received hot-rolled plate, which are presented as the pole figures in Figure 3-2a measured using synchrotron x-ray diffraction (S-XRD). The details of the annealing tests, grain size measurements based on the linear intercept method, and texture measurement using S-XRD were already presented in previous chapters.

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 Figure 3-1. The angle,  $\theta$ , 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  $10^{-3}\text{s}^{-1}$ . 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  $\pm 0.5\%$ .

### **5.2.2 Polycrystal VPSC model with the H-P relationships**

In the VPSC model, it is assumed that each grain with the distinct orientation can be described as an ellipsoidal inclusion embedded in a homogeneous effective medium (HEM) [30, 31]. The strain and interactions with the HEM are heterogeneous for each grain, while the averaged behavior over all the grains is the same as the macroscopic boundary condition, which fulfilled the self-consistent manner by plastic deformation.

In the present study, the tangent self-consistent scheme was used to describe the linearization of the grain-medium interaction [5] and the contribution of the twinning to the grain reorientation was represented by the predominant twin reorientation (PTR) scheme proposed by Tome *et. al* [32]. In the PTR scheme, a threshold value of the twin volume fraction is initially set by the user. At each deformation step, the accumulated twinned volume fraction is compared to this threshold limitation for each grain. If it exceeds the set threshold value, the grain is reoriented to the new direction following the predominant twin system in that grain.

According to the crystal plasticity theory, Voce hardening is employed to describe the hardening responses of each deformation system,  $\alpha$ , by increasing the critical resolved shear stress,  $\hat{\tau}^\alpha$  (instantaneous CRSS) as a function of accumulated shear strain in the grain,  $\gamma_{ac}$ :

$$\hat{\tau}^\alpha = \tau_0^\alpha + \frac{k^\alpha}{\sqrt{D}} + (\tau_1^\alpha + h_1^\alpha \gamma_{ac}) \left[ 1 - \exp\left(-\frac{h_0^\alpha}{\tau_1^\alpha} \gamma_{ac}\right) \right] \quad 5-2$$

,where  $\tau_0$ ,  $h_0$ ,  $h_1$ , and  $\tau_0 + \tau_1$  are the initial CRSS, the initial hardening rate, the asymptotic hardening rate and the back-extrapolated CRSS, respectively.  $k^\alpha$  is the grain size sensitivity of a deformation mode,  $\alpha$ , and  $D$  is the grain size.  $\tau_0$ ,  $h_0$ ,  $h_1$ , and  $\tau_0 + \tau_1$  are the single crystal parameters for VPSC modeling that describe the crystal plasticity for each deformation mode. The parametric approach was used for the simulations by iteratively fitting to room temperature mechanical test data and texture evolutions for specimens with different initial textures and grain sizes. The best fitting results are presented in Table 5-3.

The aim of this work is to provide advances in the simulation of the complex combination effects of grain size and texture in Mg alloy and provide the accurate hardening parameters.

Table 5-3. Constitutive parameters of basal, prismatic, and pyramidal slips as well as extension twin for visco-plastic self-consistent modeling (VPSC) with the Hall-Petch relationships, which can describe the stress-strain behaviors and texture evolution of the specimens with the various grain sizes ranging from 26 to 78 $\mu\text{m}$ .

| <b>Deformation modes<br/>(MPa)</b> | <b><math>\tau_0</math></b> | <b><math>\tau_1</math></b> | <b><math>h_0</math></b> | <b><math>h_0</math></b> | <b><math>h^*B</math></b> | <b><math>h^*P</math></b> | <b><math>h^*Py</math></b> | <b><math>h^*T</math></b> | <b><math>k_\tau</math></b> |
|------------------------------------|----------------------------|----------------------------|-------------------------|-------------------------|--------------------------|--------------------------|---------------------------|--------------------------|----------------------------|
| Basal <a>                          | 7                          | 4                          | 2550                    | 70                      | 1                        | 1                        | 1                         | 1                        | 86                         |
| Prismatic <a>                      | 16                         | 27                         | 2700                    | 300                     | 1                        | 1                        | 1                         | 1                        | 186                        |
| Pyramidal <c+a>                    | 40                         | 80                         | 4000                    | 62                      | 1                        | 1                        | 1                         | 1                        | 260                        |
| Extension Twin                     | 6                          | 0                          | 0                       | 0                       | 1.2                      | 1.7                      | 1                         | 1                        | 116                        |

### 5.3 Results

#### 5.3.1 *Tensile behaviors with different grain size and texture*

The tensile stress-strain behaviors for four different grain sizes at five different loading orientations ( $\theta=0, 22.5, 45, 67.5$ , and  $90^\circ$ ) were measured through tensile tests (solid line) and simulated using VPSC modeling (open symbol) in Figure 5-1(a-e), respectively. The concave-up stress-strain curves for tension along 0 and 22.5 degrees indicate the activation of extension twin during the deformation. On the other hand, the common concave-down curves for the tension along 45, 67.5 and 90 degrees imply the slip dominant deformation mechanisms. Therefore, it is revealed that deformation modes change with the LD, and therefore the initial texture, which was discussed in earlier chapters. Moreover, the stress-strain behaviors for the specimens with different grain sizes show the similar shapes for a given LD,  $\theta$ , which indicate that the dominant deformation mode does not change significantly with the increase in the grain size up to  $78\mu\text{m}$ . However, the hardening behaviors are different with the increase in grain size. For example, the hardening rates for the specimens with grain size at  $43\mu\text{m}$  is higher than those for the ones with the grain size at  $26\mu\text{m}$  after the yielding for  $0^\circ$ ,  $22.5^\circ$ , and  $45^\circ$  cases.

Moreover, the differences between the stress-strain curves obtained from simulation and experiment are shown in Figure 5-1 for different grain sizes and loading orientations.

Figure 5-1. Tensile stress-strain behaviors of the specimens with four different grain sizes ranging from 26 to 78 $\mu\text{m}$  as a function of the angle  $\theta$  between the LD and ND obtained through both experiment (solid line) and VPSC simulation (symbol) with the constitutive parameters presented in Table 5-3 as well as the difference curves between the two: (a)  $\theta=0^\circ$  loading case; (b)  $22.5^\circ$ ; (c)  $45^\circ$ ; (d)  $67.5^\circ$ ; and (e)  $90^\circ$ .

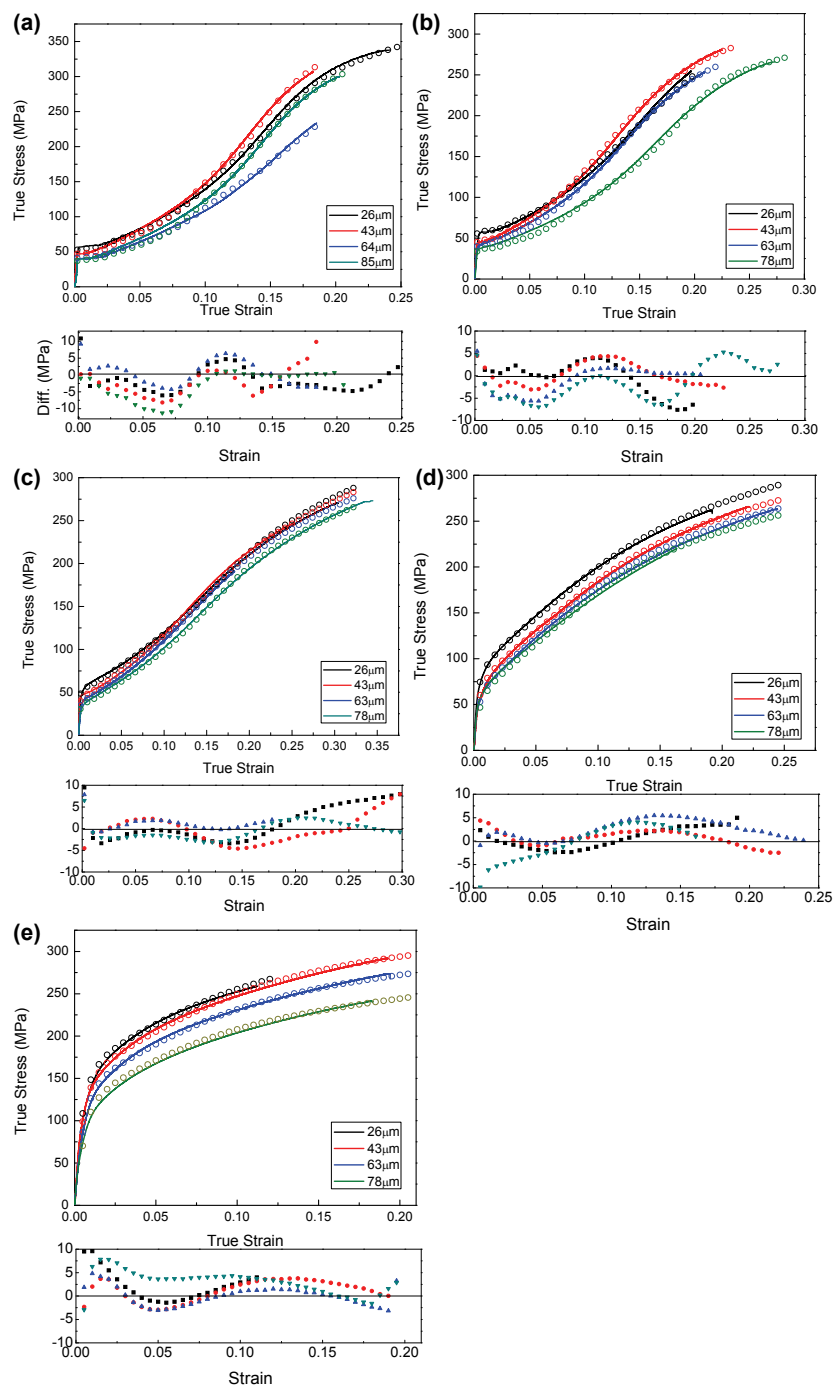


Figure 5–1 Continued

It is shown that the maximum difference is about 5MPa (less than 1.5%) by comparing the VPSC simulation results with the experiment results, which indicates that the grain size effect on both yielding and hardening behaviors can be simulated accurately through the VPSC modeling with the incorporation of the H-P relationships. The effect of grain size on the hardening behaviors may be the results of the change of relative activation of various deformation modes with the increase in grain size for a given initial texture, and it will be discussed in detail later.

### ***5.3.2 Activation of deformation modes as a function of grain size with different initial textures***

Figure 5-2 (a-e) represents the relative activation of various deformation modes as a function of strain for 0, 22.5, 45, 67.5 and 90° cases, respectively, when the grain sizes are 26 $\mu\text{m}$  and 78 $\mu\text{m}$ . Various deformation modes dominate the deformation with the changes in the LD and therefore, initial textures. For the 0° tension case when the grain size is 26 $\mu\text{m}$ , extension twin is the main deformation mode to accommodate the strain at the small strain stage, followed by the prismatic slip in the daughter grains. When the strain is higher than 0.15, pyramidal slip is activated, since the stress is high enough. Meanwhile, some basal slip is activated to accommodate the strain when the strain is 0.15-0.225. When the grain size is at 78 $\mu\text{m}$  for the same LD case, the relative activation curves of various deformation modes does not change qualitatively. However, the relative activities of pyramidal slip (0.230) and basal slip (0.120) are a little bit higher than the ones (pyramidal: 0.211 and basal: 0.085) with the grain size with 26 $\mu\text{m}$ .

Figure 5-2. Relative activities of various deformation modes, including basal, prismatic, and pyramidal slip as well as the extension twinning, (solid lines) and volume fraction of extension twinning (open symbols) as a function of strain for the tension along different loading orientations respects to the normal direction (ND) of the Mg plate,  $\theta$ , simulated through VPSC modeling for the specimens with the grain size as 26 $\mu\text{m}$  and 78 $\mu\text{m}$ . (a)  $\theta=0^\circ$ ; (b)  $\theta=22.5^\circ$ ; (c)  $\theta=45^\circ$ ; (d)  $\theta=67.5^\circ$  and (e)  $\theta=90^\circ$ .



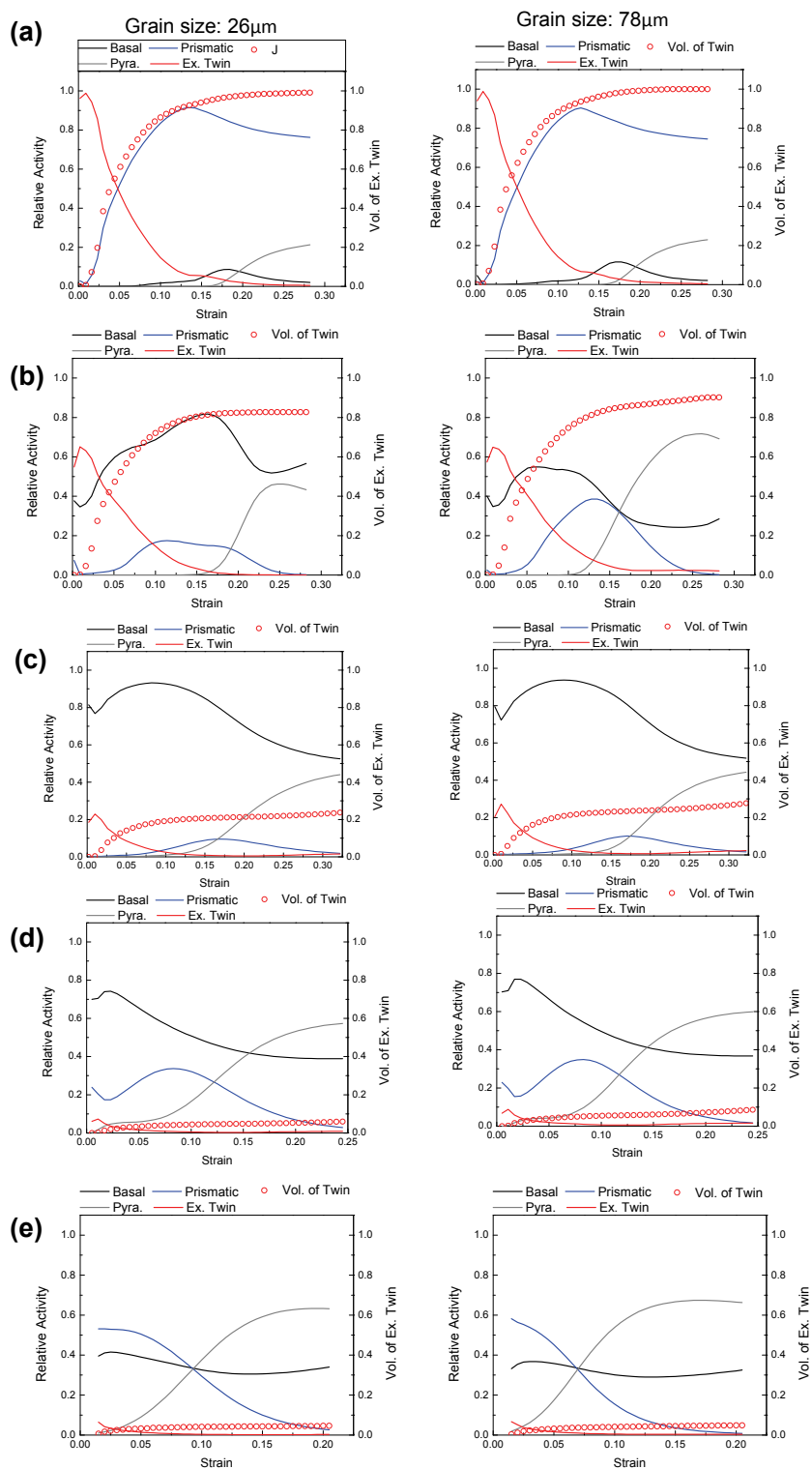


Figure 5-2 Continued

For the 22.5° tension case when the grain size is 26μm, extension twin and basal slip are the two possible deformation modes which compete with each other at the small strain. The volume fraction of twin increases up to the 0.829 with the strain due to the activation of extension twinning. In the daughter grains, both basal and prismatic slips are activated to accommodate the strain. When the strain is higher than 0.15, the pyramidal slip is activated. When the grain size is at 78μm, the relative activities show the similar trend with some quantitative differences. First, more prismatic slip is activated to accommodate the strain by replacing the basal slip at the daughter grains. Moreover, the volume fraction of extension twin (0.904) is higher than that when the grain size is 26μm. Finally, the pyramidal slip starts earlier (at the strain as low as 0.115) and has higher activities with the increase of the grain size, which indicates that the pyramidal slip is easier to be activated when the grain size is bigger.

For the tension along 45°, the basal slip is dominant during the deformation, and gradually replaced by the extension twinning when the strain is higher than 0.125. Moreover, some extension twin is activated as a supplemental mode at the beginning of the plastic deformation. With the increase in strain, more extension twin occurs and higher twin volume fraction is shown. Also, a little more pyramidal slip is activated at higher strain compared to the ones observed in other LD cases. The relative activities for the tension along 67.5° case are very close to those for the 45° case.

For the tension along 90° case, the prismatic slip is the most active mode at the beginning of the plastic deformation and gradually replaced by the pyramidal slip mode

during the deformation. When the grain size is as high as  $78\mu\text{m}$ , the pyramidal slip increases at the higher rate with the increase in strain.

### **5.3.3 Texture evolution with the change of LD**

With the input parameters of VPSC simulation with the H-P relationships, the texture evolution during the plastic deformation can also be accurately simulated. The final textures of the specimens with various LDs when the grain size is  $26\mu\text{m}$  measured from the S-XRD and VPSC simulation are represented as pole figures in Figure 5-3(a-e), respectively. The textures obtained through the VPSC simulation are very close to the ones obtained from the S-XRD. However, the simulated textures are much sharper than the measured cases, which is common for VPSC simulations. The error may come from the approximation of HEM scheme [33]. It is shown that the texture has more significant change during the deformation when  $\theta$  is lower than  $45^\circ$  due to the activation of extension twinning. The extension twinning results the crystal rotation of about  $86^\circ$  [34], which rotates the  $\{0002\}$  pole from the ND (center of the pole figure) to the TD-RD plane (circumference of the pole figure). The subsequent deformation modes change after the activation of extension twin due to the changes in the crystal orientation. For the  $\theta$ s higher than  $45^\circ$ , the slip systems are the preferred deformation modes with limited extension twin. Therefore, the texture does not change significantly during the deformation. The textures stay the same with the increase in the grain size for all five LD cases. Therefore, only the textures of specimens with the grain size of  $26\mu\text{m}$  for various LDs are shown to verify the accuracy the VPSC simulation in this chapter.

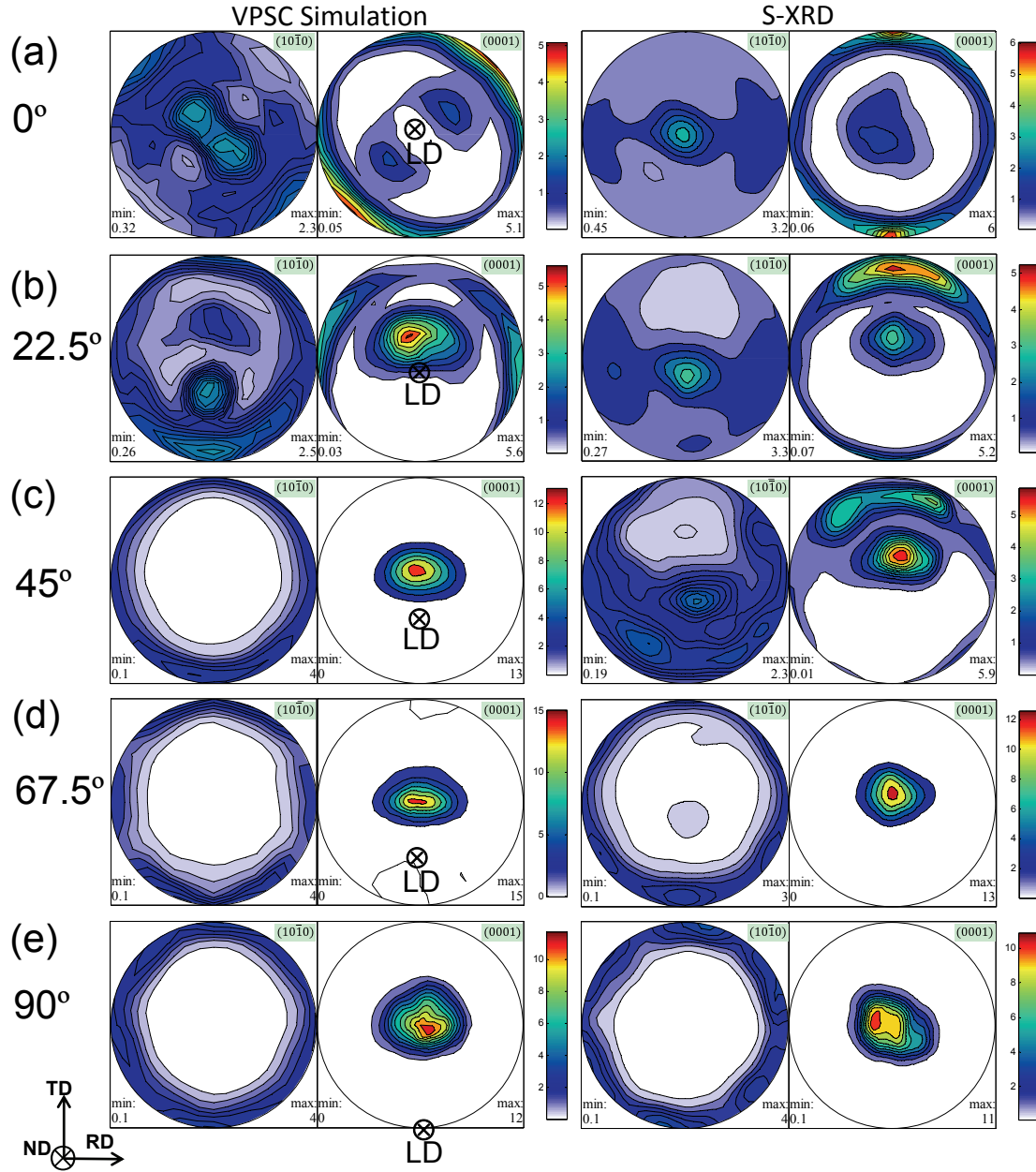


Figure 5-3.  $\{10\bar{1}0\}$  and  $\{0001\}$  pole figures (PFs) of deformed specimens with the grain size of  $26\mu\text{m}$  for the tension along different loading orientations with respects to the normal direction (ND) of the Mg plate,  $\theta$ , obtained through VPSC modeling and S-XRD measurements. (a)  $\theta=0^\circ$ ; (b)  $\theta=22.5^\circ$ ; (c)  $\theta=45^\circ$ ; (d)  $\theta=67.5^\circ$  and (e)  $\theta=90^\circ$ .

#### 5.3.4 Grain size effect on the flow stress with various LD

The 0.1 and 0.15 offset flow stresses as a function of  $D^{-1/2}$  for the various LDs are shown in Figure 5-4 (a-b), respectively. It is indicated that the flow stresses and the square root of grain size do not have a linear relationship. Moreover, the 0.10 and 0.15 offset flow stresses for tension along 0 and 22.5° show the “abnormal” increase with the increase in the grain size from 26 ( $D^{-1/2}=6.20\text{mm}^{-1/2}$ ) to 43 $\mu\text{m}$  ( $D^{-1/2}=4.83\text{mm}^{-1/2}$ ). The 0.10 offset flow stress for tension along 0° increase with the increase in grain size from 65 to 78 $\mu\text{m}$  as well. On the other hand, the flow stresses decrease with the increase in the grain size in the other stages. However, the slopes of the flow stress as a function of  $D^{-1/2}$  keeps decrease with the increase of  $D^{-1/2}$  for all five LDs cases, which indicates that the grain size sensitivity for flow stress is not constant for a given  $\theta$  with the changes in grain size. For instance, the slope ( $\text{MPa}\cdot\text{mm}^{1/2}$ ) decreases from 58.8 to 5.9 to 16.7 with the increase of  $D^{-1/2}$  ( $\text{mm}^{-1/2}$ ) from 3.57 to 3.98 to 4.83 for the tension along 90° case. It is shown that the grain size sensitivity is higher when the grain size is smaller for each LDs case. The reason of the “abnormal” increase of the flow stress with the increase in grain size and the slope changes will be discussed in details later.

Moreover, the 0.15 flow stresses show the similar trend with the increase of  $D^{-1/2}$  compared to the 0.10 flow stress curves for various LDs cases. However, the slopes are more or less larger than those of the 0.10 flow stresses.

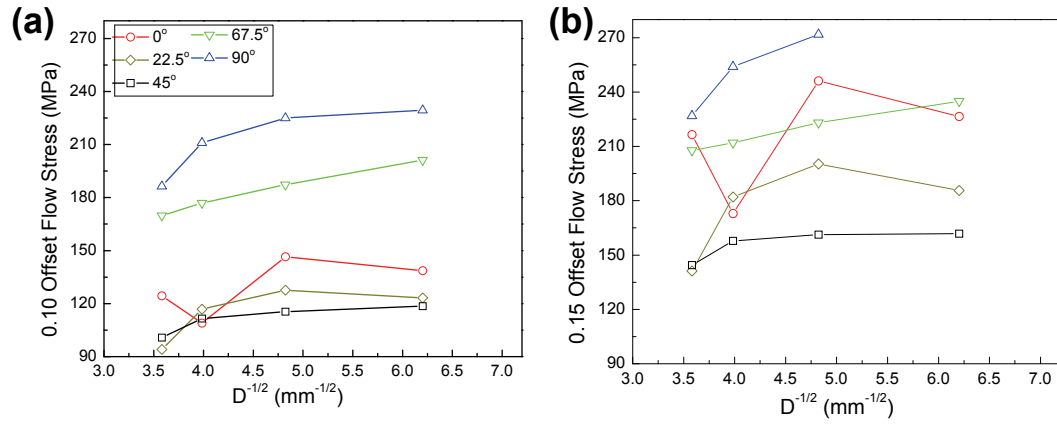


Figure 5-4. The 0.10 and 0.15 offset flow stresses as a function of grain size,  $D^{-1/2}$ , with different loading orientations,  $\theta$ , between the LD and ND from the experiment.

## 5.4 Discussion

### 5.4.1 *The input parameter of VPSC simulation with the incorporation of the H-P relationship*

VPSC simulation is employed to investigate the changes in activation of deformation mechanisms with the changes in the initial textures and their influence on the texture evolution. In order to understand the grain size effect on the mechanical behavior, deformation modes and texture evolution during the plastic deformation, grain size sensitivity,  $k^\alpha$  for each deformation mode are included in the input parameters. As mentioned earlier,  $\tau_o$  for each deformation mode was not the “real” critical resolved shear stress. Actually, it is the resolved shear stress for a given grain size. In the current study, the “real” critical resolved shear stress with the grain size sensitivity,  $k^\alpha$ , for each deformation mode were used. Therefore,  $\tau_o$  for each deformation mode is lower than the ones used earlier studies. It is worth noting that other hardening parameters including  $h_o$ ,  $h_1$ , and  $\tau_1$  for all deformation modes remain the same as before.

The  $\tau_o$  and  $k^\alpha$  for the deformation mode,  $\alpha$ , which can fit the stress-strain behaviors of different grain size and LD cases show good agreements with H-P study from the experiment listed in

Table 5-2. For the slip systems including the basal, prismatic, and pyramidal slip,  $\tau_o$  for basal slip (7MPa) is the lowest, while the one for pyramidal slip (40MPa) is the highest and that for prismatic slip (16MPa) is in between. Moreover, the grain size sensitivity  $k^\alpha$  for each deformation mode shows the similar trend. The  $k^\alpha$  (MPa· $\mu\text{m}^{1/2}$ ) for basal slip is as low as 86 and that for pyramidal slip is more than 3 times of that (300).

The  $k^\alpha$  ( $\text{MPa}\cdot\mu\text{m}^{1/2}$ ) for prismatic slip is 186, which is in between. The grain size sensitivity can be explained through the dislocation pile-up theory. Therefore, the  $k^\alpha$  is proportional to the CRSS and Burgers vector of the dislocation, which can explain the same trend between the  $\tau_o$  and  $k^\alpha$ .

The  $\tau_o$  for extension twin is slightly smaller than that of the basal slip, while the  $k^\alpha$  is higher than the basal slip. 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 [35], twin is generated as a result of the microslip concentrations due to the dislocation pileup, and it was proposed that  $k_\tau^{\text{Twin}} \approx (1.5 - 7)k_\tau^{\text{Slip}}$  in bcc metals. In the current study, it was found that  $k_\tau^{\text{Twin}} = 1.35k_\tau^{\text{Basal}}$ , which is close to the lower boundary of the relationship between the twinning and slip systems identified by Armstrong.

#### ***5.4.2 The grain size effect on the activation of deformation modes***

For the tension along  $45^\circ$  case, the basal slip is the dominant deformation mode due to the highest Schmid factor among all possible modes. However, the extension twin can also be activated due to the lower CRSS compared to the basal slip and the angle distribution of the initial texture. Therefore, the basal slip and extension twin are the two competitive modes at the beginning of the plastic deformation. Figure 5-5a shows the activation of extension twin as a function of strain for the grain size 26, 43, 65,  $78\mu\text{m}$  for the tension along  $45^\circ$  case. It is shown that the activity of extension twin increases from



the 0.185 to 0.205 with the increase in the grain size from 26 to 78 $\mu\text{m}$  when the strain is 0.002 and the maximum value increases from 0.228 to 0.273 when the strain is 0.010.

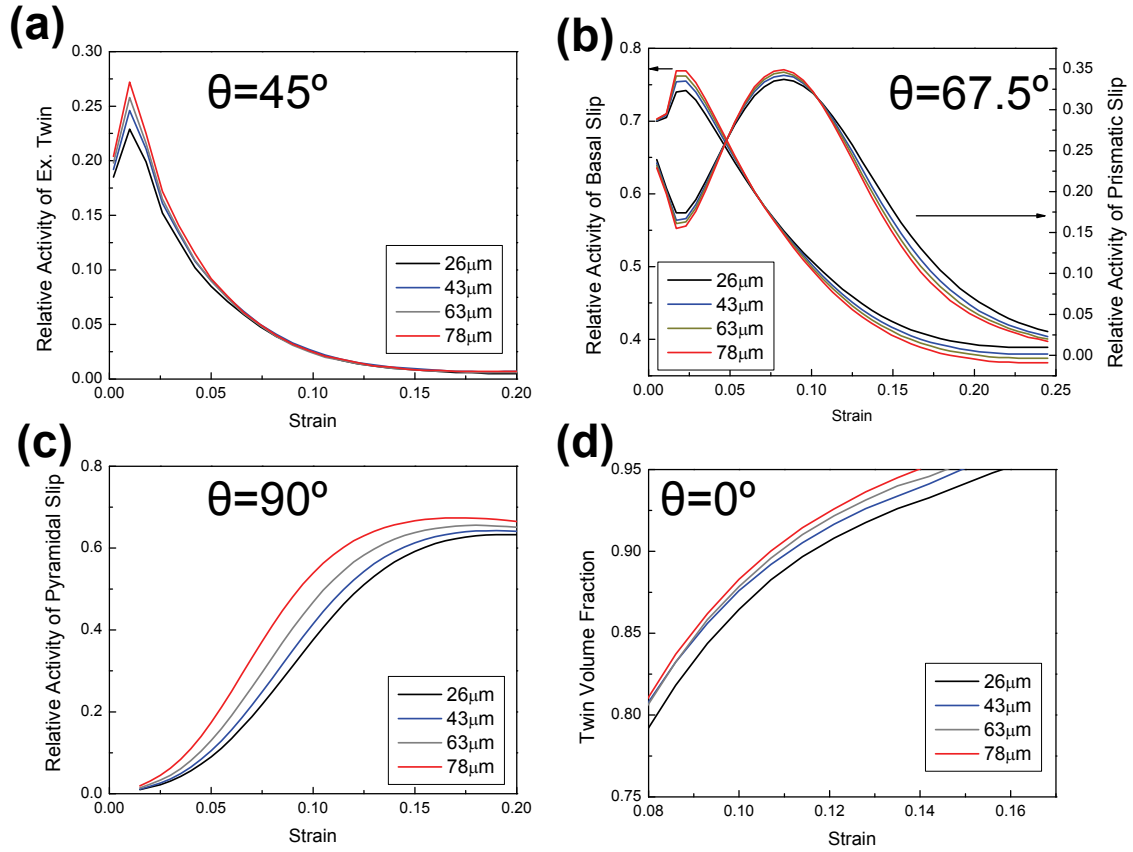


Figure 5-5. The relative activities of various deformation modes and volume fraction of extension twin evolutions with the increase in the grain size from 26 $\mu\text{m}$  to 78 $\mu\text{m}$ : (a) the relative activity of extension twin as a function of strain with various grain size for tension along 45° case, (b) the relative activity of basal and prismatic slip as a function of strain with various grain size for tension along 67.5° case, (c) the relative activity of pyramidal slip as a function of strain with various grain size for tension along 90° case,

and (d) the volume fraction of extension twin as a function of strain with various grain size for tension along  $0^\circ$  case.

Therefore, it is illustrated that more extension twin is activated in the bigger grain size when competing with the basal slip. Considering the grain size sensitivities of basal slip and extension twin,  $k_t^{\text{Twin}} = 1.35k_t^{\text{Basal}}$ , the resolved stress of extension twin decreases more rapidly than that of basal slip with the increase in grain size, which leads to the higher activation of extension twin at bigger grain sizes.

For the tension along  $67.5^\circ$  case, basal and prismatic slips are the two possible deformation modes at lower strains. Figure 5-5b show the relative activities of basal and prismatic slips as a function of strain for various grain sizes. It is revealed that the basal slip shows the lower activity while the prismatic slip is more active in the bigger grain sizes due to the higher  $k^\alpha$  for prismatic slip than that for the basal slip. When the strain is higher than 0.15, the activities of both basal and prismatic slip decrease with the increase in the grain size due to the activation of pyramidal slip, which is shown more clearly in the tension along  $90^\circ$  case.

The pyramidal slip gradually replaces the prismatic slip as the strain increases in this case. Figure 5-5c shows the activation of pyramidal slip with various grain sizes as a function of strain at  $90^\circ$  tension case. It shows clearly that the pyramidal slip increases much faster with the increase of the strain in the bigger grain size and the activity is higher as well, which is the result of the higher  $k^\alpha$  for pyramidal slip as well. Therefore, it is indicated that the higher activation of the deformation mode with the higher grain size sensitivity,  $k^\alpha$ , at the bigger grain size case is expected.

#### ***5.4.3 The effect of changes in activation modes on the flow stress due to the changes in grain size***

The activation of the deformation mode with larger grain-size sensitivity is relatively higher with the decrease in the grain size, which makes the flow stress increase faster. Therefore, the slope of the flow stress decreases with the increase in  $D^{-1/2}$  for the various LDs, Figure 5-4. For instance, more extension twin occurs for the tension along  $45^\circ$  with the increase of grain size by replacing the basal slip. More prismatic slips are activated for the  $67.5^\circ$  case by replacing the basal slip and more pyramidal slip are activated for the  $90^\circ$  case by replacing the prismatic slip, which all cause the slopes to decrease faster with the increase in the  $D^{-1/2}$ .

On the other hand, Figure 5-5d shows the twin volume fraction with the increase in the strain from 0.08 to 0.17 for the tension along the  $0^\circ$  for various grain sizes. As mentioned earlier, the activation of extension twin is higher in the larger grain size when it competes with the basal slip. Therefore, the volume fraction of the extension is highest when the grain size is  $78\mu\text{m}$ . It is demonstrated that the stress increases fast when the extension twin is activated due to the texture hardening effect. Therefore, the 0.10 and 0.15 offset flow stresses increase when the grain size increases from 26 to  $43\mu\text{m}$  and causes the “abnormal” H-P relationship to occur for the tension along 0 and  $22.5^\circ$  cases, which is illustrated in Figure 5-4.

## 5.5 Conclusions

The influence of the grain size on the flow stress, in terms of the effects on the activation of various deformation modes are studied through the visco-plastic self-consistent (VPSC) simulation with the incorporation of Hall-Petch (H-P) relationships using a commercial hot-rolled AZ31B Mg alloy plate with a strong  $\{0001\} \langle 10\bar{1}0 \rangle$  plane texture. The texture variations are facilitated by changing the angle  $\theta$ , between the uniaxial tensile loading direction (LD) and the normal direction (ND) of the plate to manipulate the activation of various deformation modes. The conclusions are as follows.

1. The stress-strain behaviors and texture evolutions for various LDs, and therefore initial texture and grain size can be simulated using the VPSC simulation with the incorporation of the H-P parameter,  $k^\alpha$ , for various deformation modes. Different deformation modes are activated with the changes in the initial textures. Extension twinning followed by the prismatic slip is the dominant deformation mode for the tension along  $0^\circ$ . Basal slip is the dominant mode for tension along  $45^\circ$ , while the prismatic slip is the most preferred deformation mode and gradually replaced by the pyramidal slip as the strain increases for the tension along  $90^\circ$  case. Tension along  $22.5^\circ$  and  $67.5^\circ$  cases are the combination of  $0^\circ$  and  $45^\circ$  cases or the  $45^\circ$  and  $90^\circ$  cases, respectively. The pyramidal slips are activated when the strain is high enough for all five LDs.
2. The relative activities of various deformation modes for each LD case remain qualitatively the same with the increase in the grain size. However, the activities change quantitatively. For the  $45^\circ$  case, more extension twins are activated at the bigger grain size by replacing the basal slips. Higher activity of prismatic slip is shown when the grain

size becomes larger by replacing the basal slip for the 67.5° case, while more pyramidal slip by competing with the prismatic slip. It is demonstrated that the deformation mode with the higher grain size sensitivity,  $k^\alpha$ , has the relatively higher activity when the grain size is larger.

3. The effect of grain size on the flow stresses is studied in terms of the activation of deformation modes as a function of LDs,  $\theta$ . For the slip dominant cases ( $\theta=45$ , 67.5, and 90° cases), the slope of the flow stress as a function grain size  $D^{-1/2}$  decreases with the increase of the  $D^{-1/2}$ , which indicates that the grain size sensitivities increase with the decrease in the grain size due to the activation of deformation modes with higher  $k^\alpha$  at the larger grain size. For the tension along 0 and 22.5° cases, the “abnormal” decrease in the flow stress was observed with the decrease in the grain size. Considering the higher activation of extension twin and higher volume fraction of twinning at the larger grain sizes, stronger texture hardening is expected, which provides the higher stress.

4. In the current study, the “real” critical resolved shear stresses (CRSSes) are used as  $\tau_o$  instead of the resolved shear stresses for a given grain size. The  $\tau_o$  for basal slip (7MPa) is the lowest, while the one for pyramidal slip (40MPa) is the highest and that for prismatic slip (16MPa) is in between. Moreover, the  $k^\alpha$  (MPa· $\mu\text{m}^{1/2}$ ) for basal slip is 86 and that for pyramidal slip is up to 300, which is more than 3 times than that of the basal slip. The  $k^\alpha$  (MPa· $\mu\text{m}^{1/2}$ ) for prismatic slip is 186. The H-P relationships for the slip systems obtained from the VPSC simulation show good agreements with the ones calculated through the dislocation pile-up theory. The  $\tau_o$  for extension twinning (6MPa) is slightly lower than that for basal slip, while the  $k^\alpha$  is slightly higher than that for basal

slip ( $116 \text{ MPa} \cdot \mu\text{m}^{1/2}$ ), which indicate that grain size effect of extension twin is different from the slip systems.

## Chapter 6

### **Effect of extension twinning on the hardening behavior in a Mg alloy: comparison between the a-axis compression and c-axis tension**

The effects of the  $\{10\bar{1}2\}$  extension twinning on the microstructure, texture, and deformation mechanisms in a wrought Mg alloy are studied through the comparison between two most twinning favorable loading paths: tension along normal direction (ND) and compression along transvers direction (TD) of the hot-rolled Mg plate. The synchrotron x-ray diffraction (S-XRD) and electron backscatter diffraction (EBSD) measurements were used to study the texture and microstructure evolutions introduced by the twinning. The visco-plastic self-consistent (VPSC) modeling was also used to investigate the activation of various deformation mechanisms during the plastic deformation. A single set of input crystal constitutive parameters were verified through iterative comparisons to the stress-strain behavior and texture evolution during uniaxial tension and compression at various loading orientations between the ND and TD. The model is, then, used to describe the tension along ND and compression along TD cases by only varying in the latent hardening parameters,  $h^{twin,slip}$ .

The stress-strain behaviors, texture, and microstructure evolutions and the activation of various deformation modes are compared between the tension along ND case and compression along TD case. Moreover, the effect of twin variant selection on the texture and twin morphology evolution is discussed. Finally, the effect of the

microstructure and texture evolution introduced by the extension twin on the hardening behaviors is discussed in terms of texture hardening, Hall-Petch hardening, and Basinski hardening. The texture hardening is discussed based on the Schmid factor calculations and VPSC simulation, while the Hall-Petch like hardening is quantitatively investigated by observing the changes in the mean free path of prismatic  $\langle a \rangle$  and basal  $\langle a \rangle$  dislocations in the twinned daughter grains during  $c$ -tension and  $a$ -compression, respectively. Finally, the Basinski hardening effect is discussed for the  $a$ -compression case, where the prismatic dislocations activated in the matrix are later become sessile due to dramatic orientation change in the daughter grains.



## 6.1 Introduction

Mg alloys are promising structure metals for applications in the communication and automotive fields due to the low density and high specific strength [1]. However, the application of the wrought Mg alloys is limited due to the low formability and high anisotropy even though they have superior mechanical properties compared to the cast Mg alloys [2]. After a thermo-mechanical processing, a strong texture generally forms with their most of the basal planes aligned parallel to the process (shear) direction [3, 4]. For instance, the basal planes are almost parallel to the extrusion direction of the hot-extruded bars and parallel to the rolling direction of the hot-rolled plate [5].

It clearly shows that  $\langle a \rangle$  dislocation slips along basal, prismatic, or pyramidal planes provide only a total of four independent slip systems and none of them are capable of accommodating any plastic strains along the crystallographic  $c$  direction. The activation of second pyramidal  $\langle c+a \rangle$  slip system provides additional degrees of freedom needed for an arbitrary isochoric plastic strain, and has been confirmed experimentally [6]. On the other hand, deformation twinning can also provide the needed  $c$ -axis strain component for generalized deformation. In general, the strain contributed by twinning is much smaller than that by the slip. However, the twin can also provide indirect contributions to the strain through grain reorientation, release of strain concentration, and trigger other types of dislocations, all of which provide dramatic influences on the plastic behavior of Mg alloys [7]. However, the twinning also gives rise to significant anisotropy in plasticity [8]. The twin boundaries, as an obstacle, inhibit the movement of dislocations, which provides the Hall-Petch-like hardening effect [9]. In Mg alloys, the

prismatic or basal slip can be hindered by the  $\{10\bar{1}2\}$  twin, which raises the local stress concentration around the twin boundaries by the dislocation pile-up [10]. If the dislocations around the interface can form the mobile dislocation in the twin, the stress can be released. On the other hand, the high stress concentration may alternatively lead to the crack initiation. The stress concentration may become a site for the nucleation of conjugate twins or formation of crack, depending on the cleavage strength of metal. If the fracture strength is much higher than the twin nucleation, the twin interface may become the effective sites for nucleation of other twins. On the other hand, the crack can be initiated at or near the twin interfaces [6, 11, 12].

The work of twin can only be positive, which means that the twin can only provide the strain in one direction so that the twin is sensitive to the loading (strain) path. Since the  $c/a$  ratio is close to 1.624 for Mg alloys, the  $\{10\bar{1}2\}$  extension twinning can be activated when the  $c$ -axis is stretched [12]. Therefore, two loading paths are the most favorable for the activation of the extension twinning: compression perpendicular to the  $c$ -axis and the tension along the  $c$ -axis. However, almost all of the research in the literature [13-15] on the extension twinning is limited to the compression perpendicular to the  $c$ -axis (e.g., compression along rolling direction and compression along extrusion direction since the basal planes are mostly parallel to the processing direction of a wrought Mg alloy). It was reported that the texture and microstructure evolutions are distinctly different between the  $a$ -axis compression and  $c$ -axis tension due to the difference in twin variant selections [16]. It was also pointed out that it would lead to

different deformation characteristics including the flow stress and strain hardening rate, which are related to the twin-twin or twin-dislocation interactions [17, 18].

The interactions of twin with dislocations with  $\langle a \rangle$ ,  $\langle c \rangle$ , or  $\langle a+c \rangle$  Burgers vectors have been studied. The twin interfaces can also be the sources of  $\langle c+a \rangle$  dislocations. The interaction of  $\langle a \rangle$  dislocations with  $\{10\bar{1}2\}$  twin can produce the  $\langle c+a \rangle$  dislocation and the partial dislocation on the twin interface by the reaction:  $\frac{a}{3}\langle\bar{2}110\rangle_B = \frac{a}{6}\langle11\bar{2}3\rangle_{TP} - b_T$ . In this case, the interaction of twin with dislocations will improve the plasticity of Mg when non-basal slip is activated [19].

It was found that the dramatic reduction in the twin propagation rate when two intersecting twins grew equally in a grain, which is the results of the twin-twin interaction [20]. The twin-twin interaction comes from the decrease in twin propagation and growth rate with the increase in the twin nucleation rate. In fact, twin nucleation stress is higher than the propagation according to the general assumptions based on the fundamentals of twinning mechanisms. The twin propagation is accommodated by the twin dislocation gliding. If dislocations could easily form and glide along the twin boundaries, stress can be relax. Therefore, the nucleation is suppressed and the twin propagation and growth are enhanced. However, if the twin dislocation gliding is substantially impeded by impingements against impenetrable obstacle twins, the local stress increases dramatically due to the stress concentration. Therefore, it can be concluded that twin penetration stress is even higher than the twin nucleation stress, which inhibits the twin propagation and growth when the twin intersects with another twin variant, which is the one effect of the twin-twin interaction.

Another type of twin-twin interaction is the Hall-Petch effect. When narrow twin band forms and intersected with the twins with other variants, the dislocation mean-free path reduces.

The effects of twin variant selection on the twin-twin and twin-slip interactions under two loading paths have not been quantitatively investigated. Moreover, the activation of slip systems in the parent and daughter grains is different since the orientation relationships between the loading direction and the orientations of parent/daughter grains are different even though both of these two loading paths are the twin favorable cases. For instance, considering that the extension twin introduces the  $86.3^\circ$  crystal reorientation, for the *c*-axis tension case, the daughter grains are under tension perpendicular to the basal pole after twinning, while, for the *a*-axis compression case, the daughter grains are under compression along basal poles after twinning. Therefore, the activation of slip systems is distinctly different under these two loading scenarios, which may provide different twin-slip interactions and cause different hardening behaviors. Therefore, the investigation on the effect of extension twinning on the texture and microstructure evolution by comparing the *c*-axis tension and *a*-axis compression cases will shed lights to the fundamental understanding of the twin-twin and twin-slip interactions, and, in turn, the anisotropy in hardening mechanisms.

In summary, the twin has three main mechanisms that change the plasticity of Mg alloys [7, 21-28].

(1) The  $86.3^\circ$  crystal reorientation introduced by the activation of extension twinning generates the significant texture evolution, which changes the flow stress and

hardening behavior significantly [12, 16]. The changes in the crystal lattice orientation to either a harder or softer orientation, which is named as texture softening or hardening effect. The crystal reorientation generally causes the texture “hardening” in the Mg AZ 31 alloy since the daughter grains always end up in the harder orientations [14, 27].

(2) The Hall-Petch-like hardening effect originates from the reduced mean free path for dislocations within the twinned daughter grains. It was reported that the twin boundary blocks the movement of the dislocation slip so that the higher strain-hardening effect is expected with the decrease in the effective grain size. Mahajan [29] found that the deformation twin inhibits the movement of dislocation in the face centered cubic (fcc) crystals, which explains the increase in the strain-hardening rate by the decrease in the effective grain size after the twinning occurs. Moreover, in some crystal plasticity models, the effect of twinning on the hardening behaviors was taken into account by assuming that the twin boundaries inhibit the dislocation movement following Hall-Petch effect to achieve good fits to the observed mechanical responses of titanium and zirconium alloys [30]. However, Chichili [31] reported that after the low temperature deformation of Ti, the yield strength does not increase significantly compared with the one without the preloading even though the preloading introduced high twin density in the Ti. Therefore, it is also indicated that the twin does not always provide hardening effects.

(3) The strain hardening due to a glissile-to-sessile transition of dislocations, already present in the region experiencing the twinning shear transformation, is called Basinski hardening mechanism [32]. It was proposed that the glissile dislocations activated before twinning are changed to the sessile condition as a result of twinning

shear transformation. Therefore, the daughter becomes harder than the parent. Salem [33] verified the Basinski mechanism by a comparison of the microhardness tests in the twin and parent regions. It was found that overall, the hardness of daughter is 30% harder than the matrix in the pure Ti, while the hardness of the thick twin is not much difference compared with the thin one, which indicated the effect of the extension twin on the hardening came from the Basinski effect instead of the Hall-Petch-like effect in this case.

The relation between the twin and fracture are complicated. Concerning the association of fracture with twinning, it is stated that twinning and fracture are both essentially cataclysmic processes of strain relief, and the conditions which are ideal for the development of one will generally be satisfactory for the development of the other. This competitive role of twinning against cracking depends closely on the character of the material and defect properties and the experimental conditions [12].

In this chapter, we will first present the stress-strain behaviors under the compression along transverse direction and tension along the normal direction of the hot rolled Mg plate. Throughout the chapter, TD, RD, ND, and LD refers to the transverse, rolling, and normal direction of the plate, as well as the loading direction (tension or compression), respectively. Also, the compression along TD is the compression perpendicular to the  $c$ -axis since the strong plane texture in the hot-rolled Mg plate, which will be called “ $a$ -compression” as a simplification, while the tension along ND is the tension along  $c$ -axis, which will be called “ $c$ -tension”. Then, we will analyze the texture and microstructure evolutions through synchrotron x-ray diffraction (S-XRD) and electron backscatter diffraction (EBSD). Finally, the activation of various deformation

modes will be simulated through the visco-plastic self-consistent (VPSC) model for the  $c$ -tension and  $a$ -compression cases. Moreover, the twin variant selections under these two loading paths will be discussed in terms of the Schmid factor. In addition, the effect of twin variant selections on the microstructure and texture evolution will be discussed. The difference in the slip activities for both parent and daughter grains under these two loading paths will also be discussed. Finally, the role of extension twin on the plastic deformation will be summarized using the following factors: (1) the crystal reorientation, which introduces the texture evolution and alters the subsequent deformation modes; (2) the twin boundaries hinder the dislocation movements, which is the Hall-Petch-like hardening effect; and (3) the twin-dislocation interaction, which changes with the distinct texture and microstructure evolutions under these two loading paths.

The current study provides a basic understanding of the influence of twin variants on the microstructure and texture evolution, which provides the insights to understanding of the twin-twin and twin-slip interactions, and therefore, the effect of the twin on the hardening behaviors.

## **6.2 Experimental details**

### **6.2.1 Material**

The material used in this study is a commercial AZ31B Mg alloy, which was hot rolled with O-temper annealing condition with the initial equi-axial grain size of  $26\pm 3.5\mu\text{m}$ . The dimension of the rolled plate is  $400\times 250\times 90\text{mm}$ , which is adequate for the tension test along the normal direction of the plate. The rolled Mg plate has strong

plane texture with most of the  $c$ -axis of the crystal parallel to the ND of the plate as shown in the pole figures measured using synchrotron x-ray diffraction (S-XRD), Figure 6-1.

### **6.2.2 Mechanical testing**

A series of dog-bone tensile specimens with the LD parallel to the ND and cylindrical compression specimens with the LD parallel to TD were prepared by electrical discharge machining (EDM) from the rolled Mg plate, Figure 6-1a. Mechanical tests were performed using an Instron 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  $10^{-3}\text{s}^{-1}$ . The gauge dimensions of the tensile specimen are 3.0 mm wide, 1.6 mm thick, and 16.5 mm long. The tests were stopped at the strains of 0.005, 0.02, 0.03, 0.05, 0.075, 0.103, 0.127, 0.150, and at the fracture. The texture and microstructure were measured using ex-situ post-mortem S-XRD and EBSD. The dimension of the cylinder compression specimens are 8mm in length, 6mm in diameter. Compression tests were stopped at the strains of 0.005, 0.02, 0.03, 0.05, 0.075, 0.103, and fracture. Several repeat measurements were performed for both tensile and compression tests, which showed an excellent reproducibility with a variation in the flow stress within  $\pm 0.5\%$ .



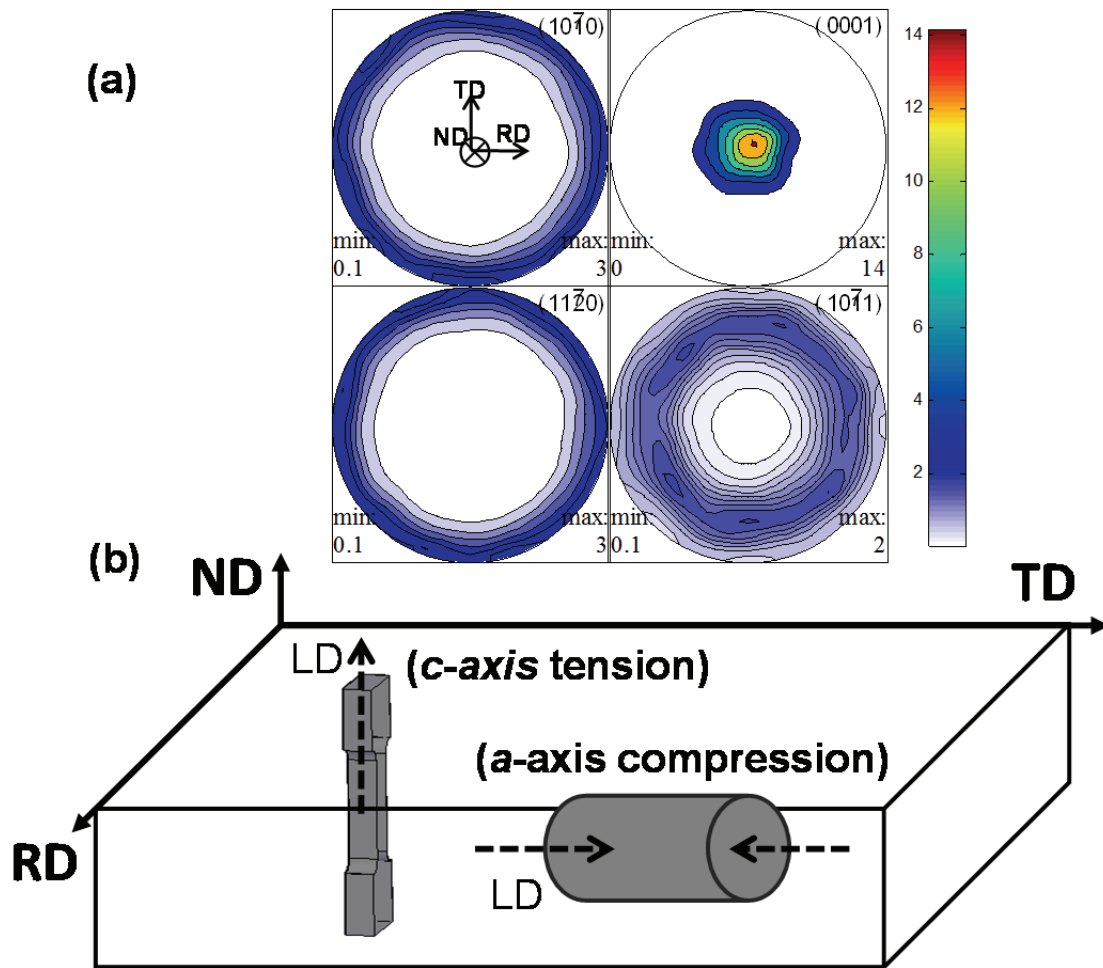


Figure 6-1 Initial texture of as-received AZ31B Mg alloy rolled plate and a schematic illustrating the preparation of tensile and compressive specimens: (a)  $\{10\bar{1}0\}$  and  $\{0001\}$  pole figures (PFs) measured using S-XRD, and (b) tensile test with the loading direction parallel to the ND (named as *c*-tension) and compressive test with loading direction parallel to the TD (named as *a*-compression).

### **6.2.3 Texture measurements**

The crystallographic texture was measured using synchrotron x-ray diffraction (SXRD) at beamline 11-ID-C, Advanced Photon Source (APS), Argonne National Laboratory. The wavelength was 0.107841 Å, and the nominal distance between the sample and the 2D detector was set as 1540 mm, which covered 22 reflections (01.0 to 03.2) for the hcp structure. The beam size was 600 μm × 600 μm, and the cross section of the samples used for the texture measurement was 3 mm × 3 mm. 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  $\omega$  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 [34] for texture presentation in terms of pole figures. Further details of the texture measurement can be found in [35].

### **6.2.4 EBSD measurements**

EBSD installed in a field emission scanning electron microscope was used to identify the active twin variant and developed texture. Automated EBSD scans were performed in the stage control mode with TSL data acquisition software on an area of 0.12 mm<sup>2</sup> with a step size of 0.3 μm. The specimens for the EBSD examination were

ground on 2400 grit silicon carbide papers and polished using 1  $\mu\text{m}$  diamond paste, followed by a final polish with colloidal silica to remove strains and provide a high quality surface finish. Analysis of the EBSD data was accomplished with TSL OIM analysis software, and the data with a confidence index  $>0.1$  were used for texture and twin analysis

### **6.3 VPSC modeling approach**

The stress-strain behaviors, texture evolutions, and activation of different deformation modes were simulated by the VPSC modeling. In the VPSC model, it is assumed that each grain was described as an ellipsoidal inclusion embedded in a homogeneous effective medium (HEM) [36, 37]. The strain and interactions with the HEM are different for each grain, while the averaged behavior over all the grains is the same as the macroscopic boundary condition, which fulfilled the self-consistent manner by plastic deformation. The VPSC model simulation provides a detailed description of the anisotropic plastic behavior of Mg alloys, which has the HCP crystal structure and limited deformation systems.

In the present study, the tangent self-consistent scheme was used to describe the linearization of the grain-medium interaction [38] and the contribution of the twinning to the grain reorientation was represented by the predominant twin reorientation (PTR) scheme developed by Tome [39, 40]. In the PTR scheme, a threshold value of the twin volume fraction is initially set by the user. At each deformation step, the accumulated twinned volume fraction is compared to this threshold limitation for each grain. If it

exceeds the set threshold value, the grain is reoriented to the new direction following the predominant twin system in that grain. The details of the VPSC simulation was already described the previous chapters.

According to the crystal plasticity theory, Voce hardening is employed to describe the hardening responses of each deformation system,  $\alpha$ , by increasing the critical resolved shear stress,  $\hat{\tau}^\alpha$  (instantaneous CRSS) as a function of accumulated shear strain in the grain,  $\gamma_{ac}$ :

$$\hat{\tau}^\alpha = \tau_0^\alpha + (\tau_1^\alpha + h_1^\alpha \gamma_{ac}) \left[ 1 - \exp\left(-\frac{h_0^\alpha}{\tau_1^\alpha} \gamma_{ac}\right) \right] \quad 6-1$$

,where  $\tau_0$ ,  $h_0$ ,  $h_1$ , and  $\tau_0 + \tau_1$  are the initial CRSS, the initial hardening rate, the asymptotic hardening rate and the back-extrapolated CRSS, respectively. Here,  $\tau_0$ ,  $h_0$ ,  $h_1$ , and  $\tau_0 + \tau_1$  are the input single crystal parameters for VPSC modeling that describe the crystal plasticity for each deformation mode. In the current study, four deformation systems including:  $\{0001\} \langle 11\bar{2}0 \rangle$  basal slip;  $\{\bar{1}010\} \langle 11\bar{2}0 \rangle$  prismatic slip;  $\{10\bar{1}1\} \langle 11\bar{2}3 \rangle$  pyramidal slip, and  $\{10\bar{1}2\}$  extension twin are taken into consideration to accommodate the plastic deformation in the AZ31 Mg alloy at room temperature. The hardening parameters including the  $\tau_0^\alpha$ ,  $h_0^\alpha$ ,  $h_1^\alpha$ ,  $\tau_0^\alpha + \tau_1^\alpha$  and latent hardening  $h^{\alpha\beta}$  used in the current VPSC simulation for the tension and compression cases are listed in Table 6-1.

Table 6-1. Critical resolved shear stress (CRSS) and hardening parameters used for the VPSC modeling.  $\tau_0$ ,  $h_0$ ,  $h_1$ , and  $\tau_0 + \tau_1$  are the initial critical resolved shear stress (CRSS), the initial hardening rate, the asymptotic hardening rate and the back-extrapolated CRSS, respectively.  $h^*$  is the latent hardening parameter. Two different sets of latent hardening parameters were used for the tension and compression (in parentheses) cases.

| <b>Deformation modes,<br/><math>\alpha</math></b> | <b><math>\tau_0</math></b> | <b><math>\tau_1</math></b> | <b><math>h_0</math></b> | <b><math>h_1</math></b> | <b><math>h^*Ba</math></b> | <b><math>h^*Pr</math></b> | <b><math>h^*Py</math></b> | <b><math>h^*Et</math></b> |
|---------------------------------------------------|----------------------------|----------------------------|-------------------------|-------------------------|---------------------------|---------------------------|---------------------------|---------------------------|
| Basal <a>                                         | 24                         | 4                          | 2550                    | 70                      | 1                         | 1                         | 1                         | 1                         |
| Prismatic <a>                                     | 52                         | 27                         | 2700                    | 300                     | 1                         | 1                         | 1                         | 1                         |
| Pyramidal <c+a>                                   | 90                         | 80                         | 4000                    | 62                      | 1                         | 1                         | 1                         | 1                         |
| Extension Twin                                    | 29                         | 0                          | 0                       | 0                       | 1.2 (0.6)                 | 1.4 (1.8)                 | 1.0 (0.8)                 | 1                         |

One set of input parameters is used for both tension and compression cases with only changes in the latent hardening parameter,  $h^{\text{twin,slip}}$ , which simulates different twin-slip interactions as a results of differences in the twin morphology evolution and activation of deformation mechanisms under these two loading cases.  $\tau_0^\alpha$  for various deformation modes were obtained through the Hall-Petch relationship study presented in Chapter 3. The input crystal orientation matrix was obtained through texture measurements using S-XRD of the as-received specimens. For each LDs, 16,240 grain orientations were modeled. In order to verify the VPSC simulation, one set of input parameter, Table 6-1, was used to simulate the stress-strain behaviors and texture evolution under uniaxial tension and compression with various loading orientations systematically changing with respect to the ND of the plate. A total of five different orientations (0°, 22.5°, 45°, 67.5°, and 90°), changing from ND to TD, for tension and three orientations (0°, 45°, and 90°) for compression were selected. It was demonstrated that this set of input crystal hardening parameters can provide a good agreement between the stress-strain behaviors and texture evolutions obtained from the experiments, which was already discussed in Chapter 4.

## 6.4 Results

### 6.4.1 Stress-strain behaviors: *a*-compression vs *c*-tension

The stress-strain curves of the *a*-compression and *c*-tension obtained from both experiments (solid lines) and VPSC simulations (open symbols) are shown in Figure 6-2.

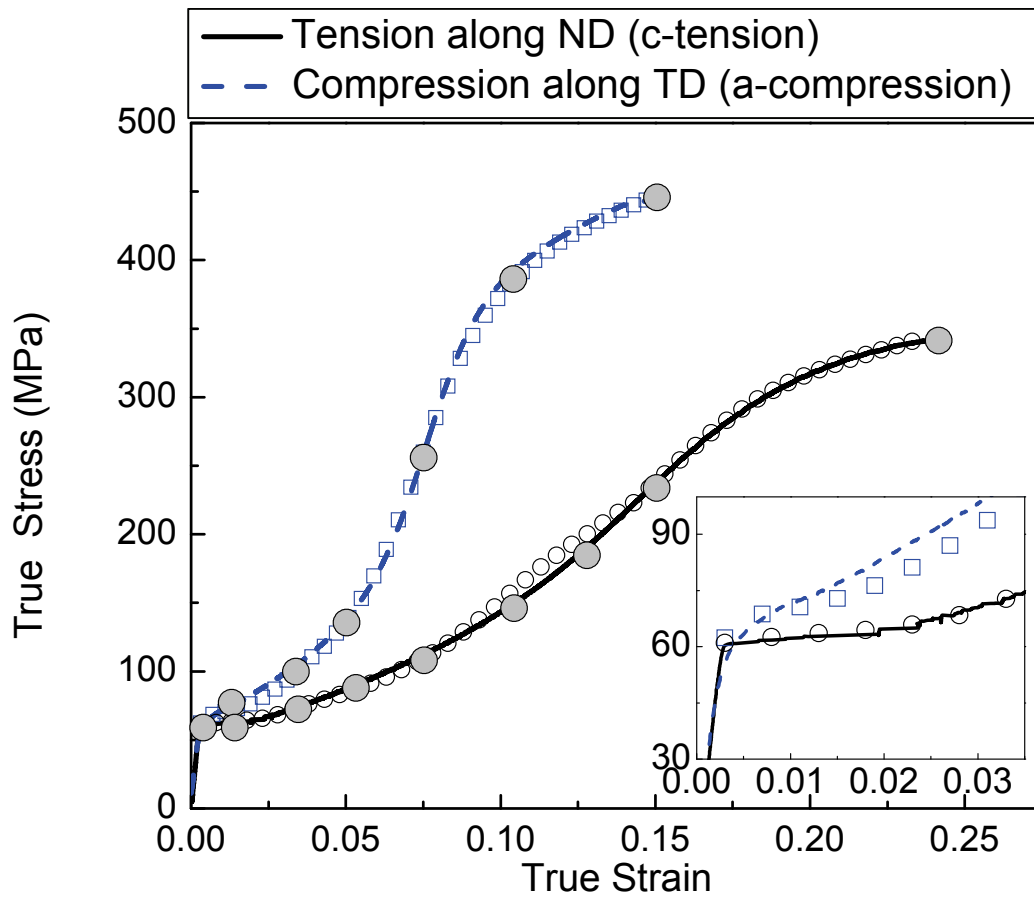


Figure 6-2 Stress-strain behaviors under tension along ND and compression along TD. The solid lines are measured stress-strain curves and the symbols are simulated using the VPSC model.

The concave-up curves for both two loading cases manifest that extension twins are prevalent during the plastic deformation. The yield strengths for  $\alpha$ -compression (61MPa) is slightly higher than that for  $c$ -tension (56MPa), while the hardening of the  $\alpha$ -compression is much higher. It is shown that when the strain is at 0.15, the flow stress for  $\alpha$ -compression is 447MPa, while that for  $c$ -tension is only 232MPa. Moreover, the ductility of the  $\alpha$ -compression (0.151) is more limited than that for the  $c$ -tension (0.241). Another difference in the stress-strain behavior between these two cases shows at the early plastic deformation stage just beyond the yield point. It is shown that the significant plateau stage appears for the  $c$ -tension case at the strain ranging from 0.003 to 0.023, where the stress almost stays the same with the increase in the strain. However, there is no obvious plateau stage under during  $\alpha$ -compression. The stress increases with the increase in the strain at this strain range, which is similar to the slip dominant deformation. This difference actually indicates that the slip system is activated in the  $\alpha$ -compression case even earlier than the activation of the extension twin, which will be discussed in details later.

The VPSC simulated stress-strain curves for both two loading paths show very good agreements with the experimental results. The characteristics of the yield behavior and flow curves are both qualitatively and quantitatively described with the input parameters used in Table 6-1. The only discrepancy is shown in the  $c$ -tension case at the strain range from 0.075 to 0.15. The VPSC simulation overestimates the stress by about 5%.



#### 6.4.2 Texture evolution

The textures are analyzed using the S-XRD and VPSC for  $c$ -tension at the strains of 0.005, 0.02, 0.03, 0.05, 0.075, 0.103, 0.127, 0.150, and 0.024 and  $a$ -compression specimens at different strains of 0.005, 0.02, 0.03, 0.05, 0.075, 0.103, and 0.125. The  $\{0002\}$  and  $\{10\bar{1}0\}$  pole figures (PFs) obtained from the S-XRD at the strain as 0.005, 0.02, 0.05 and 0.103 are presented in Figure 6-3. It was revealed that the significant texture evolutions occur during the plastic deformation in both cases. The intensity of the  $\{0002\}$  component at the center (ND) in the PFs decreases with the increase in the strain up to 0.10. However, the distinctly different texture developments under these two cases were shown.

For the  $c$ -tension case, the south/north poles of  $\{0002\}$  component show up at the strain of 0.03. Subsequently, the intensities of the  $\{0002\}$  at the south/north increase rapidly with the increase in strain. More than one component intensifies during the reorientation of the  $\{0002\}$  poles from the center to the outer rim when the strain is at 0.05. Then, the homogenous rim forms in the  $\{0002\}$  PF, which means the  $c$ -axes are homogeneously distributed in the RD-TD plane at this stage with a slight orientation preference along TD (south/north pole) for the  $\{0002\}$  pole.

On the other hand, for the  $a$ -compression case, the  $c$ -axis shifts from the center to the south/north pole in the  $\{0002\}$  PF at the strain of 0.002, which is similar to the  $c$ -tension case.

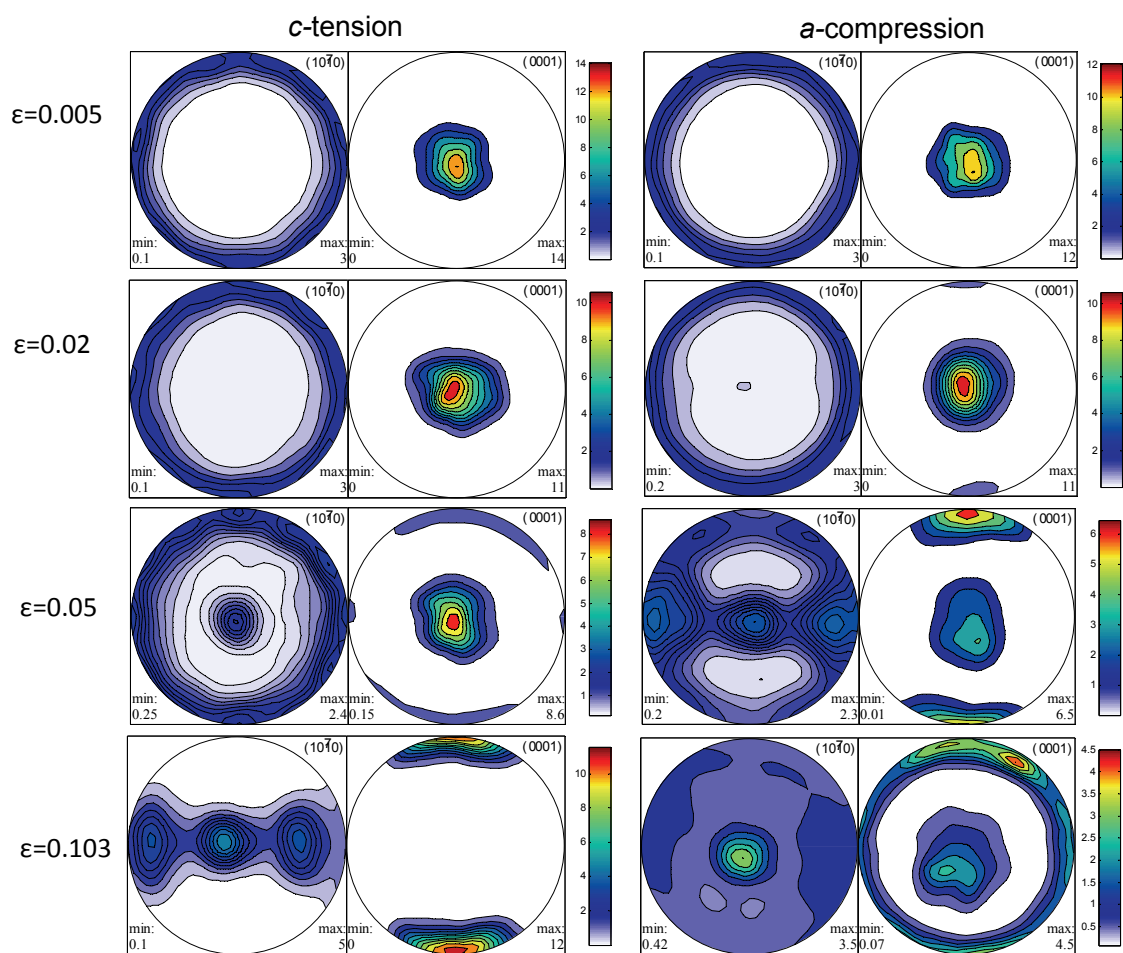


Figure 6-3  $\{10\bar{1}0\}$ , and  $\{0001\}$  pole figures (PFs) of specimens with various strain under the tension along ND (*c*-tension) and compression along TD (*a*-compression) obtained through S-XRD: (a) *c*-tension and (b) *a*-compression.

However, with the increase in strain, the intensity of the  $\{0002\}$  component at center decreases very rapidly until it fully disappears at the strain of 0.103, while  $c$ -axes in twinned regions were reorientated to a narrow range of the angles ( $<30^\circ$ ) from the TD towards the RD instead of forming the homogenous rim. It is indicated that almost no parent grains are left and almost all the  $c$ -axes of daughter grains are parallel to TD after the deformation under the current test conditions.

In addition, the texture evolutions under  $c$ -tension and  $a$ -compression obtained through the VPSC simulations are shown in Figure 6-4, which shows good agreements with the S-XRD results. The qualitative characteristics of the texture evolutions are successfully predicted, while some minor differences are evident. For instance, the texture evolutions obtained from the VPSC is more rapid and more significant than the experimental results. This may be due to the HEM approximation approach used in the current VPSC simulation as discussed in Chapter 4.

#### **6.4.3 Volume fraction of twinning**

The twin volume fraction under  $c$ -tension and  $a$ -compression cases obtained from the VPSC simulation (solid lines) and S-XRD (open symbols) are shown in Figure 6-5. It is found that the twin volume fraction increases rapidly with the increase in the strain. In the  $c$ -tension case, the extension twin starts at the very beginning of the plastic deformation and saturates at the strain of about 0.10 in both experimental and simulation results.

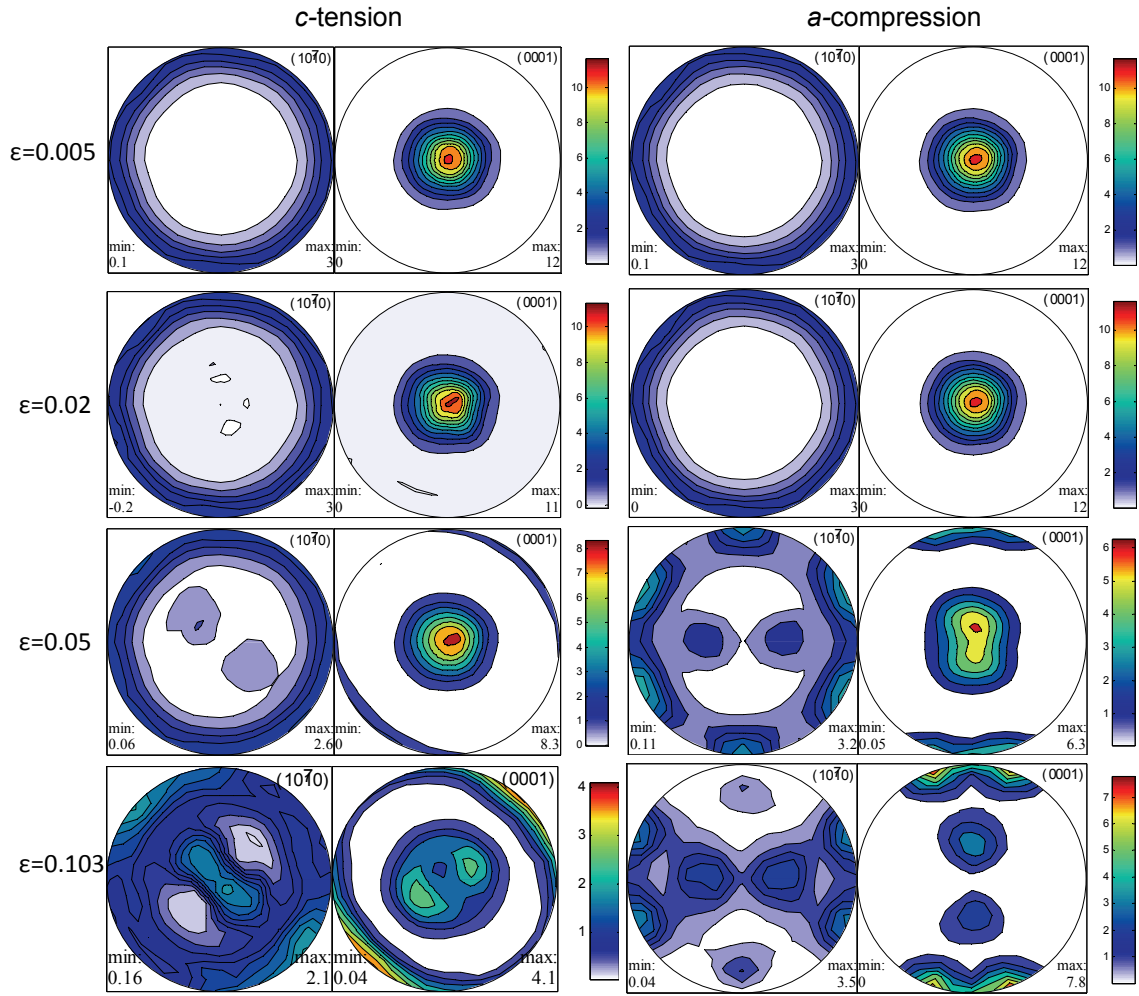


Figure 6-4  $\{10\bar{1}0\}$ , and  $\{0001\}$  pole figures (PFs) of specimens with various strain under the tension along ND (*c*-tension) and compression along TD (*a*-compression) obtained through VPSC: (a) *c*-tension and (b) *a*-compression.

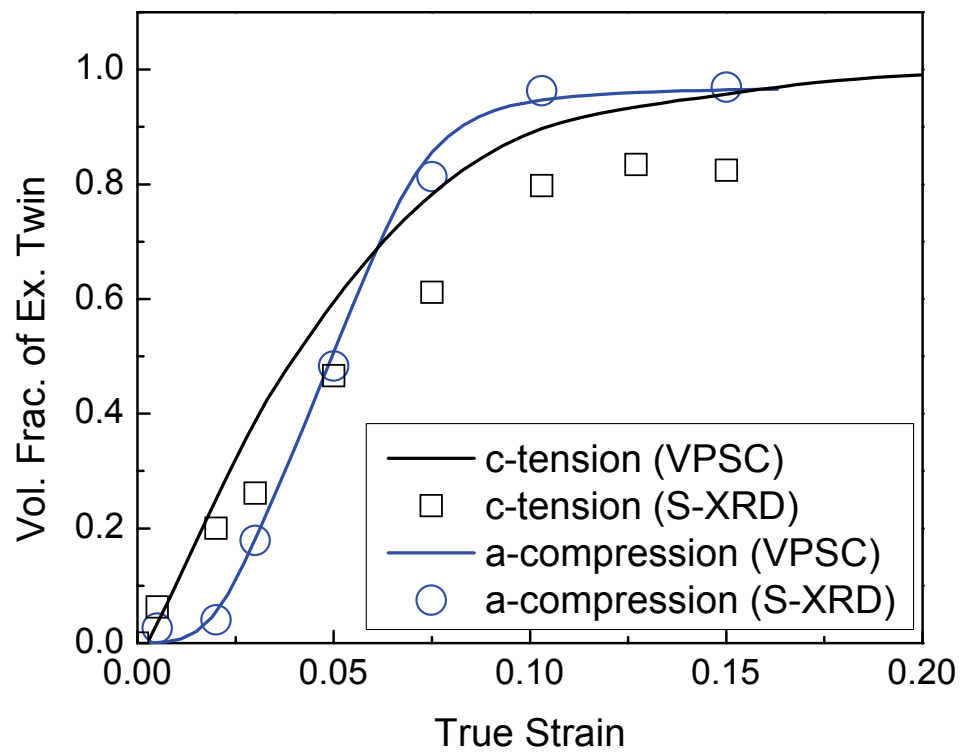


Figure 6-5 Twin volume fraction as a function of strain under the tension along ND (*c*-tension) and compression along TD (*a*-compression) obtained through VPSC simulation and S-XRD.

However, the twin volume fraction obtained from the experiment is about 10-15% lower compared to the simulation results, which may be the results of the choice of angles separating the daughter and parent grains during the intensity integration process on the pole figures measured by S-XRD. Another possible reason for the mismatch may come from the PTR assumption in the VPSC simulation. In the *c*-tension case, actually more than one variant is activated within one grain so that many twin intersections form in the matrix. Therefore, around the twin intersection points, the stress is concentrated and some subgrain boundaries are formed, which can, in turn, hinder the subsequent twin growths. Thus, some matrix may survive in these small intersection regions. However, this cannot be predicted in the VPSC simulation with the current PTR assumption, since only one variant, which is named as the predominant twin variant, is allowed in each grain. On the other hand, for the *a*-compression case, the increase in the twin volume fraction starts when the strain is at about 0.025, which is slightly higher than that in the *c*-tension case. However, once the twin occurs, the twin volume fraction increases faster than that in the *c*-tension and becomes saturated at 0.09 strain.

#### **6.4.4 Microstructure evolution: *c*-tension vs. *a*-compression**

Figure 6-6 shows the EBSD crystallographic orientation maps of the specimens deformed under *c*-tension and *a*-compression conditions at the strains of 0.005, 0.02, 0.03, 0.05, 0.075, 0.103, 0.127, 0.150, and 0.024. It is shown that the microstructure evolution is distinctly different between the two loading paths.

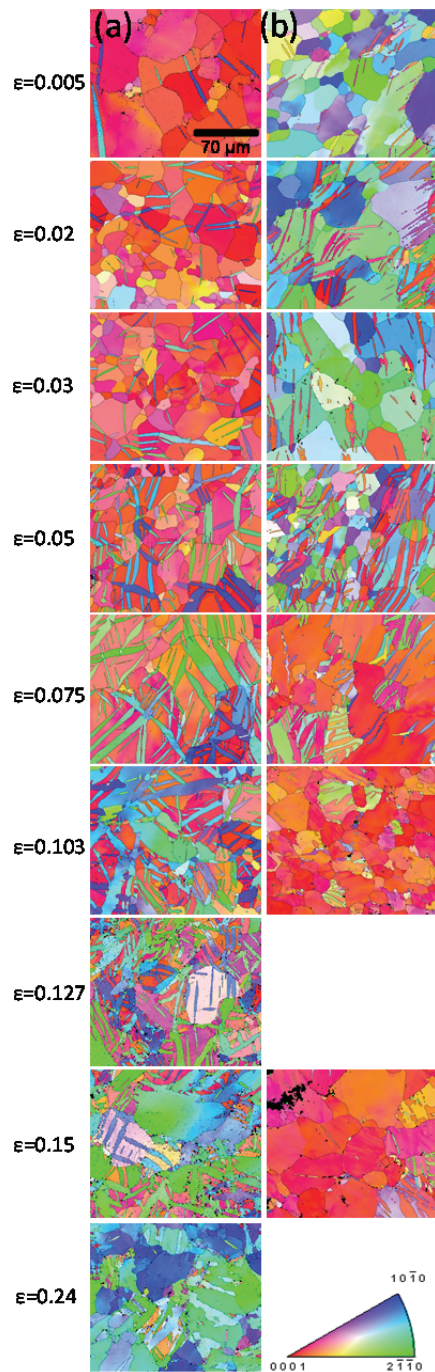


Figure 6-6 Ordination map of specimens with various strain under the tension along ND (*c*-tension) and compression along TD (*a*-compression) obtained through EBSD: (a) *c*-tension and (b) *a*-compression.

For the *c*-tension case, the blue and green parts are the twinned regions, while the red parts represent the parent. At the beginning stage of the deformation, narrow twin bands are activated in some grains with the ideal crystal orientation (i.e., *c*-axis of the crystal is parallel to the ND direction, which is shown by the red color in the orientation map). It is worth noting that the narrow twin bands are not all parallel to each other even within one grain. Two narrow twin bands encounter with each other with the misorientation angle of about 60°. With further increase in the strain, more narrow twin bands form in each grain, while the twin bands thicken as well. At about strain of 0.08, the twin bands almost covered the entire matrix region, but some small regions between twin band intersections are still in the red color, which means parent matrix regions are still left untwinned even at the large applied strain. The boundary map evolution with the increase in the strain under *c*-tension also demonstrates the twinning behaviors during the deformation, Figure 6-7a. The small angle (0-15°) boundaries, which are shown in grey color, represent the subgrain boundaries. The boundary with the angle between 15-50° is the normal grain boundary (black), while the ones between 50-70° are the twin boundaries between two different twin variants called twin-twin boundary (green). The boundaries angle that is larger than 70° is the twin-matrix boundary and marked by red color. At the beginning stage (strain <0.075), the number fraction of the misorientation at 85-90° increases very rapidly with the increase in strain. Considering the extension twinning rotates the crystal by 86.3°, these boundaries are the twin boundaries.





Figure 6-7 Boundary map with various strain under the tension along ND (*c*-tension) and compression along TD (*a*-compression) obtained through EBSD: (a) *c*-tension and (b) *a*-compression.

During the strain ranging from 0.075 to 0.103, the volume fraction of misorientation at 85-90° stays at the relatively high level with the increase in strain, which indicates that the twin boundaries do not disappear during the twin growth. When multiple twins encounter other twins with different variants, the twin boundaries cannot impregnate into the other ones, since they have different orientations. Therefore, more twin-twin boundaries appear with the misorientation angle at about 60°. This trend is summarized in Figure 6-8.

In the *a*-compression case, the red color in Figure 6-6b represents the daughter, while the blue is the parent, which is opposite to the *c*-tension case. Moreover, the parents are represented by various colors from blue to green, consistent with the homogeneous distribution of the poles in the TD-RD plane (see the orientation color map legend in Figure 6-6). It is revealed that narrow twin bands starts to form close to the high angle grain boundaries at very small strains (<0.005) for the *a*-compression case. These narrow twin bands in one grain are generally parallel to each other as only a single twin variant is dominant in this loading case. The twin bands propagate across the entire grain and the thickness of the bands also increase with the increase in the strain up to 0.075. When the strain is increased to 0.075, most of the crystal shows red color, which means almost entire region is twinned and the whole grain is reoriented. The boundary map evolution is different with the ones observed in the *c*-tension case, Figure 6-7b the twin-matrix boundaries appear at the strain from 0.02, which is the twin nucleation dominant stage, and it stays about the same with the increase in the strain up to 0.075.

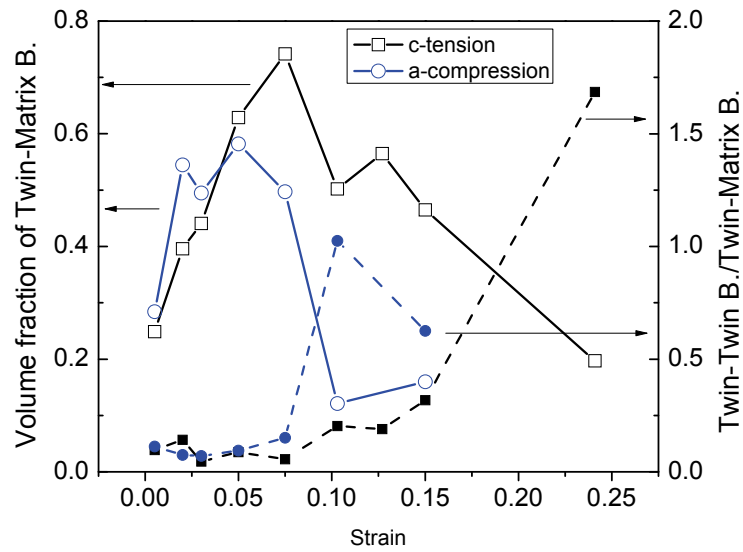


Figure 6-8 Volume fraction of twin-matrix boundary and the ratio of volume fraction of twin-twin boundary to twin-matrix boundary as a function of strain under the tension along ND (*c*-tension) and compression along TD (*a*-compression) obtained through EBSD.

When the strain is higher than 0.075, the twin-matrix boundary starts to disappear since two twins come together and become a single twinned daughter. Again, the boundary evolutions for  $a$ -compression case are also summarized in Figure 6-8.

#### **6.4.5 *The relative activities of various deformation modes as a function of strain***

Figure 6-9 shows the activities of various deformation modes and twin volume fraction evolution as a function of applied strain under  $c$ -tension and  $a$ -compression conditions obtained from the VPSC simulation.

For the  $c$ -tension case, most of the strain is accommodated by the extension twinning at the beginning of the plastic deformation. The volume fraction of twin increases until 0.08 strain. Also, prismatic slip is activated with the daughter concurrently with the increase in the twin volume fraction and the relative activity of extension twin decreases. The pyramidal slip is activated at 0.175 strain due to the much higher CRSS. The basal slip shows very limited activity for this case and the relative activities is less than 0.15 overall.

On the other hand, for the  $a$ -compression case, the prismatic slip has the highest activity at the beginning of the plastic deformation, at even earlier strain than the activation of extension twin. The relative activity of the prismatic slip is rapidly replaced by that of the extension twin when the flow strain increases from 0 to 0.02. The twin volume fraction keeps increasing until the strain is about 0.08.

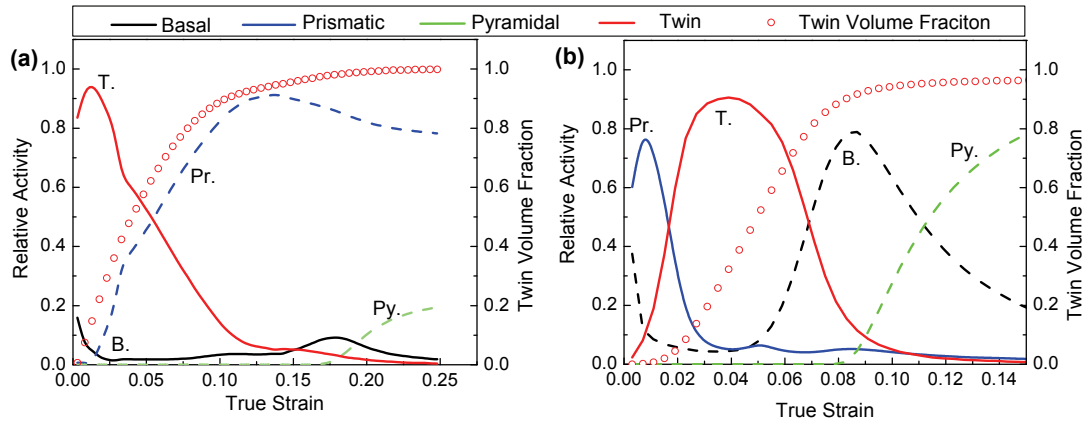


Figure 6-9 The relative activities of various deformation modes and the twin volume fraction evolution as a function of strain under tension along ND ( $c$ -tension) and compression along TD ( $a$ -compression): (a) tension along ND ( $c$ -tension), and (b) compression along TD ( $a$ -compression).

Then, the relative activity of extension twin decreases as the activation of basal slip and the twin volume fraction increases. The pyramidal slip is activated to accommodate the strain when the strain is higher than 0.08.

## **6.5 Discussion**

### **6.5.1 VPSC input parameters: *a*-compression vs *c*-tension**

VPSC simulation was employed to understand the activation of deformation mechanisms with the changes in the initial textures and their influence on the texture evolution. The parametric modeling approach was used for the simulations by iteratively fitting room temperature mechanical test data and texture evolutions. It was demonstrated that the input parameters listed in Table 6-1 can achieve good agreements in both yielding and hardening behaviors and texture evolutions under the tension with five different LDs (0°, 22.5°, 45°, 67.5°, and 90°) changing from ND to TD. Various deformation modes are activated, which generate different texture evolutions and produce a complex interplay between texture and deformation mode and influence the hardening behaviors. Therefore, the input parameters for various deformation modes including the basal, prismatic, and pyramidal slip as well as the extension twin are verified through the iterative fitting processes. The  $\tau_0$  of basal, prismatic, and pyramidal slip, as well as extension twin are obtained from the Hall-Petch relation study which presented earlier.

The simulated stress-strain behavior would show significant differences compared to the experiment results under the compression along 90° case if we use the same input hardening parameters. The simulation results would show higher flow stress at the strain

ranging from 0.10 to 0.15, while the lower flow stress at the strain from 0.02 to 0.10 than the experimental results. In order to obtain good agreements in the stress-strain behavior and texture evolutions, the latent hardening parameter,  $h^{\alpha,\beta}$ , has to be altered for the compression case. It was found that the latent hardening of extension twinning on basal and pyramidal slip needs to decrease from 1.2 to 0.6 and 1 to 0.8, respectively, while the one for prismatic slip needs to increase from 1.2 to 1.8 (Table 6-1) to achieve best results. All other input parameters remain the same as the tension case. These changes in the latent hardening of extension twinning on the slip modes are the results of the different texture and microstructure evolutions between the *c*-tension and *a*-compression cases. Therefore, the fine tuning of the latent hardening parameters provides the valuable insights to the effect of extension twin on the twin-slip and twin-twin interactions, which, in turn, affect the hardening behaviors. This will be discussed in detail in Section 6.5.6.

### 6.5.2 Variant selection of extension twin under two loading paths

The  $\{10\bar{1}2\}$  extension twin has six variants:  $(10\bar{1}2)[\bar{1}011]$ ,  $(01\bar{1}2)[0\bar{1}11]$ ,  $(\bar{1}102)[1\bar{1}01]$ ,  $(0\bar{1}12)[01\bar{1}1]$ ,  $(\bar{1}012)[10\bar{1}1]$ ,  $(1\bar{1}02)[\bar{1}101]$ . Three of them,  $(10\bar{1}2)[\bar{1}011]$ ,  $(01\bar{1}2)[0\bar{1}11]$ ,  $(\bar{1}102)[1\bar{1}01]$ , are independent and called as V1, V2, and V3, respectively, Figure 6-10a. If we assume that the hot-rolled Mg plate has the ideal initial texture:  $(0001)[10\bar{1}0]$ , the Schmid factor of the three independent variant:  $(10\bar{1}2)[\bar{1}011]$ ,  $(01\bar{1}2)[0\bar{1}11]$ ,  $(\bar{1}102)[1\bar{1}01]$  under *c*-tension and *a*-compression can be calculated, Table 6-2. It is found that the Schmid factors for all three variants are the same (0.499) for the *c*-tension case, while that for *a*-compression are different.

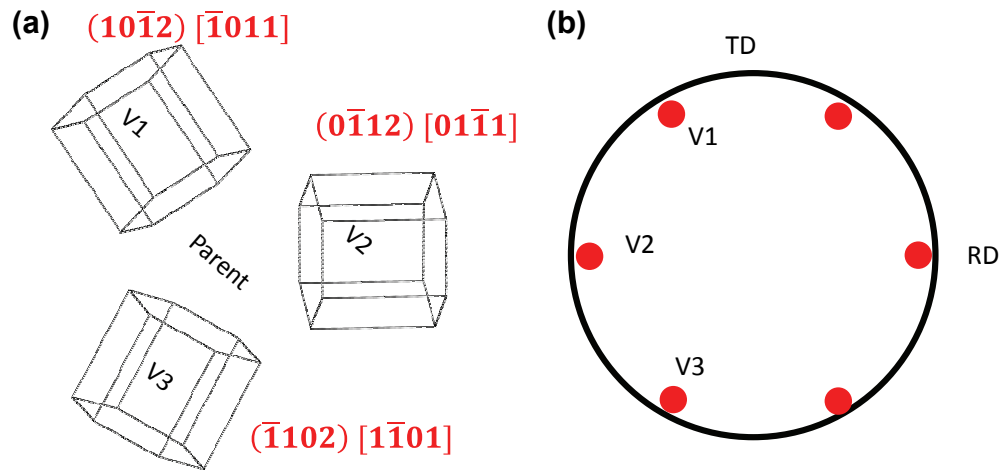


Figure 6-10 The illustration of the orientation relationship between the matrix and twinned daughter grain with different twin variant selection: (a) The illustration of the orientation relationship between the matrix and twinned daughter grain, and (b) the illustration of twin orientation in the  $\{0002\}$  pole figures.



Table 6-2. Ideal Schmid factors,  $m$ , calculated for basal slip, prismatic slip, pyramidal slip, and extension twin as a function of the loading orientation,  $\theta$ , between the LD and ND for uniaxial tension and compression cases. The ideal Schmid factors presented here are the maximum value for each case calculated assuming a perfect  $\{0001\} \langle 10\bar{1}0 \rangle$  plane texture. The twin variants; V1, V2, and V3; represent  $(10\bar{1}2)[\bar{1}011]$ ,  $(01\bar{1}2)[0\bar{1}11]$ ,  $(\bar{1}102)[1\bar{1}01]$ , respectively.

| Loading Path                                 |          | Basal | Prismatic | Pyramidal | Extension Twin |       |       |
|----------------------------------------------|----------|-------|-----------|-----------|----------------|-------|-------|
|                                              |          |       |           |           | V1             | V2    | V3    |
| Tension // ND<br>( <i>c</i> -tension)        | Parent   | 0     | 0         | 0.446     | 0.499          | 0.499 | 0.499 |
|                                              | Daughter | 0.056 | 0.431     | 0.357     | -              | -     | -     |
| Compression //TD<br>( <i>a</i> -compression) | Parent   | 0     | 0.433     | 0.446     | 0.374          | 0     | 0.374 |
|                                              | Daughter | 0.056 | 0.003     | 0.468     | -              | -     | -     |

Only  $(10\bar{1}2)[\bar{1}011]$  and  $(\bar{1}102)[1\bar{1}01]$  have the relatively higher Schmid factors (0.374), while the Schmid factor for  $(01\bar{1}2)[0\bar{1}11]$  is 0 for the  $a$ -compression case.

If the parent grain does not have the ideal orientation for  $a$ -axis (the angle between the  $a$ -axis and RD  $\phi$  ranges from 0-30°), the Schmid factors for three variants does not change with the changes in  $\phi$  because the loading orientation is parallel to the TD-RD rotation plane under  $c$ -tension. However, for the  $a$ -compression case, the Schmid factor for the variants changes significantly with the  $\phi$ . Overall, one variant shows the highest Schmid factor (higher than 0.374, which is the highest Schmid factor for the twin when  $\phi=0$ ), while other two have much lower Schmid factors.

Therefore, three different variants are selected and activated homogenously under the  $c$ -tension, while only one (or two when  $a$ -axis happens to be parallel to RD) variant is selected for the  $a$ -compression case based on the Schmid factor analysis when the parent has the  $c$ -axis parallel to the ND and  $a$ -axis has the random orientation along TD-RD plane.

### ***6.5.3 The effect of twinning on the texture and microstructure evolution under two loading paths***

The  $\{0002\}$  component of the daughter grains with different twin variants are illustrated in Figure 6-10b. It is shown that the twins with different variants have 60° misorientation with each other when the parent has the ideal orientation:  $(0001)[10\bar{1}0]$ . As mentioned above, all three variants are activated during  $c$ -tension since they have the same Schmid factors. According to the geometry relation, three  $\{0002\}$  components for

daughters which selected three variants are shown at the TD-RD rim in the PF with 60° misorientations between two neighbors.

For the *a*-compression case, the variants  $(10\bar{1}2)[\bar{1}011]$  and  $(\bar{1}102)[1\bar{1}01]$ , whose Schmid factors are relatively high, are shown at  $\pm 30^\circ$  of the TD towards the RD. The twinning with the variant  $(\bar{1}102)[1\bar{1}01]$  which should appear at the RD cannot be activated due to the low Schmid factor it has, Figure 6-10b.

If the *a*-axis of the parent has the angle,  $\phi$ , with respect to RD, the  $\{0002\}$  components of the daughter grains with three different variants will rotate around the ND with  $\phi$ . Therefore, the whole rim of the  $\{0002\}$  PF is covered homogeneously if the *a*-axes of the parent grains have the random orientations within TD-RD plane considering the Schmid factors do not change with the changes in  $\phi$  for all three variants. On the other hand, for the *a*-compression, the  $\{0002\}$  component for the daughter which chooses the variant with the highest Schmid factor (higher than 0.374) is limited in the region close to the TD within  $\pm 30^\circ$  towards the RD, which is consistent with Hong et al. [16].

Therefore, the texture evolutions obtained from the S-XRD and VPSC simulation caused by the extension twinning under the *c*-tension and *a*-compression can be explain as follows. The homogenous rim forming in  $\{0002\}$  PF after deformation under *c*-tension is consistent with the Schmid factor analysis of the extension twin variant selection, which indicates that more than one variant are selected in the parent grains. The strong south and north poles showing up for the *a*-compression case reveals that only one variant is preferred. It is worth noting that only the most preferred variant is allowed to be selected in each grain in the VPSC simulation based on the PTR scheme for the extension

twinning. However, the relatively homogenous rim in  $\{0002\}$  PF is still predicted. Since a large number of crystal orientations ( $>16000$ ) are modeled in the current simulation to describe the initial texture distribution, the parents at the global average successfully exhibit angular distributions in the twinned grains around the circumference of the PFs. For instance, the  $c$ -axes of some crystals are not perfectly parallel to the ND, which allows a different variant to be the most preferred one in the parent with different angular deviation. If the initial orientation matrix is large enough, all of the variants have the possibilities to be selected from the statistical point of view. Therefore, qualitatively, the characteristics of texture evolution under the  $c$ -tension and  $a$ -compression can be simulated accurately even though the PTR scheme have some limitations. However, the texture intensity has a shaper deviation between the preferred orientation and non-preferred orientation, since only one preferred orientation is allowed in a given “grain” in the model so that once the most preferred orientation is selected, none of the other variants can be chosen again within the same grain. This generates a sharper texture in the model simulation compared to the experimental results where more even distribution of the orientations is allowed within a single grain.

Moreover, the differences in the twin variant selections also explain the distinctly different twin morphologies develop during  $c$ -tension and  $a$ -compression. When more than one twin variants are selected during  $c$ -tension, many narrow twin bands form and many of them are not parallel with each other at the beginning of the plastic deformation. The twin bands encounter with each other and, since they are not in the same crystal orientation, the twin propagation and growth are hindered. As a result, the twin band is

relatively much narrower and the effective grain size (or dislocation mean free path) is much smaller than the initial grain size.

In the  $a$ -compression case, similar narrow twin bands form at the beginning as well. However, the twin bands are almost parallel to each other within each grain since they have the same twin variant. Therefore, very limited twin intersection is observed in this case. The twin grows by increasing the twin thickness with the increase in the strain until multiple parallel twins come together and form a wider twin band. At the end, the whole matrix reorients into the new orientation once the twin bands in the grain all connect together. Therefore, it can be considered that the twin nucleation is the dominant mode in the  $c$ -tension case, while the twin propagation and growth are more the controlling mode in the  $a$ -compression case. The different twin morphologies under these two loading paths provide different twin-slip interactions and cause the Hall-Petch like hardening effect introduced by the twin boundary.

#### **6.5.4 Deformation mechanisms under $c$ -tension vs $a$ -compression**

Table 6-2 summarizes the Schmid factor of various deformation modes under two loading paths in the matrix (parent) and twinned region (daughter) assuming the initial material has the ideal orientation  $(0001)[10\bar{1}0]$ .

For the  $c$ -tension case, it was found that the extension twin has the highest Schmid factor in the parent while both basal and prismatic slips are very hard to be activated because of their low Schmid factor. However, once twinning occurs, the

Schmid factor of the prismatic slip becomes as high as 0.433 within the daughter grains, which facilitates its high activity in the daughter grains.

However, the Schmid factor calculation in the  $a$ -compression case is different. First of all, the Schmid factor of extension twin (0.374) in the  $a$ -compression case is lower than that in the  $c$ -tension. Secondly, the prismatic slip has the highest Schmid factor (0.433) in the parent grain, while both basal and prismatic slip has the very low Schmid factors in the daughter grains.

According to the VPSC simulation, it is evident that the extension twin is the dominant deformation systems at the parent for both cases, while slip activities are very different. The most important difference is that the prismatic slip occurs earlier than the extension twin for the  $a$ -compression case during the strain lower than 0.03. Moreover, the slip activities in the daughter grains are distinctly different. In the  $c$ -tension case, prismatic is activated to accommodate the strain, while basal slip is chosen for  $a$ -compression case within the daughter, Figure 6-9.

### ***6.5.5 The effect of extension twin on the hardening behavior***

#### ***6.5.5.1 Texture hardening***

The ideal Schmid factors of the various deformation modes in both daughter and parent grains are calculated and listed in Table 6-2. It was found that the prismatic slip has the higher Schmid factor in the daughter grain in the  $c$ -tension case, while both basal and prismatic slips have the limited Schmid factor in the daughters under  $a$ -compression. Therefore, it can be concluded that the extension twinning provide the much higher

texture hardening effect in the  $a$ -compression than that in the  $c$ -tension due to the crystal reorientation. Moreover, the average Schmid factor based on the texture measurement using S-XRD and EBSD as a function of strain is presented in Figure 6-11. It was found that the changes in Schmid factor with the increase in the strain shows the similar trend as the ones obtained by assuming the initial texture has the ideal orientation. The Schmid factor of prismatic slip increases rapidly when the strain increases from 0.075 to 0.125 under  $c$ -tension, while it decrease with the increase in the strain from 0.03 to 0.08 significantly under  $a$ -compression. The average Schmid factors of various deformation modes change smoother when the deviation of grain orientations is considered. However, it is not very accurate to analyze the changes in the flow stress caused by the texture evolution by using the average Schmid factor calculation, since it does not consider any response of the neighboring grain. Therefore, the crystal plasticity modeling is required to achieve the better understanding.

According to the VPSC simulation, the texture effect introduced by the twin reorientation can be illustrated, since it considers the crystal orientation factors. Therefore, if the same input parameters for the  $c$ -tension case are used in the  $a$ -compression case, the flow stress increases dramatically from 150MPa to 500MPa with the increase in the strain from 0.08 to 0.12, Figure 6-12a. The corresponding activities of various deformation modes are shown as a function of strain in Figure 6-12b. As the activities of extension twin decreases with the increase in the strain, only basal slip can be activated to accommodate the strain.

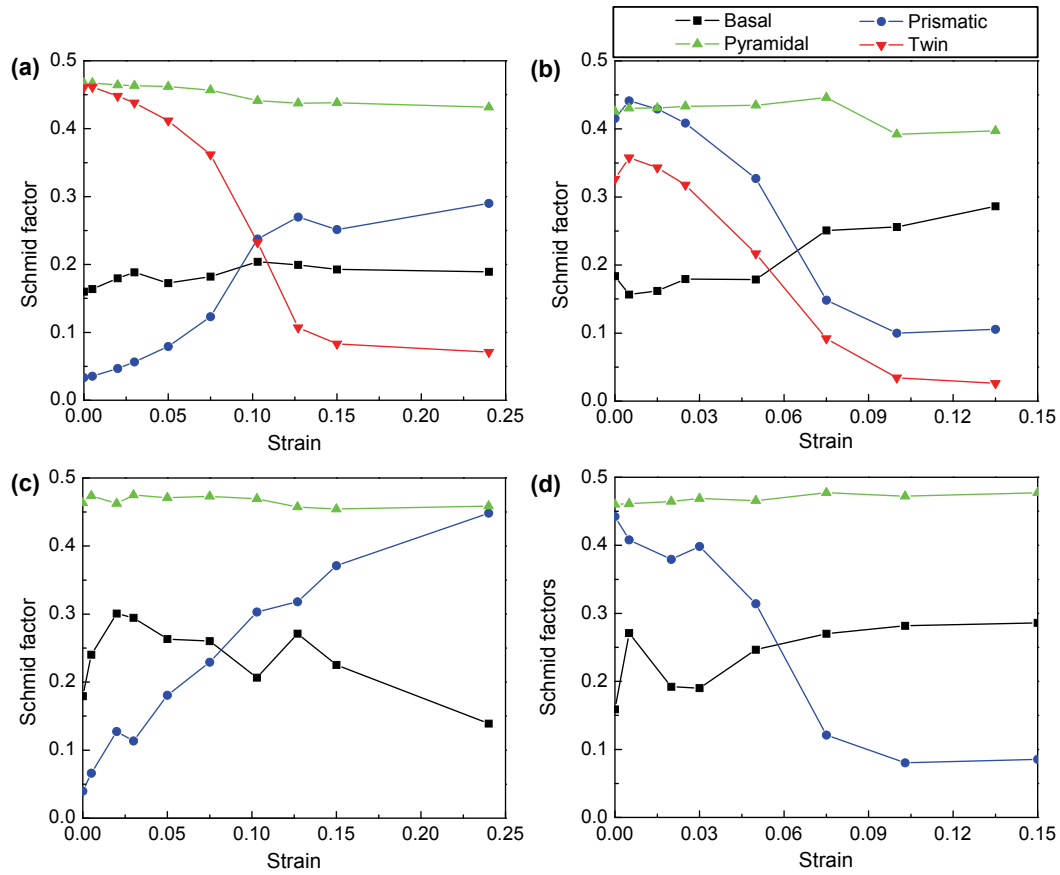


Figure 6-11 The average Schmid factor based on the orientation distribution obtained through S-XRD and EBSD under tension along ND ( $c$ -tension) and compression along TD ( $a$ -compression): (a)  $c$ -tension through S-XRD, (b)  $a$ -compression through S-XRD, (c)  $c$ -tension through EBSD, and (d)  $a$ -compression through EBSD.



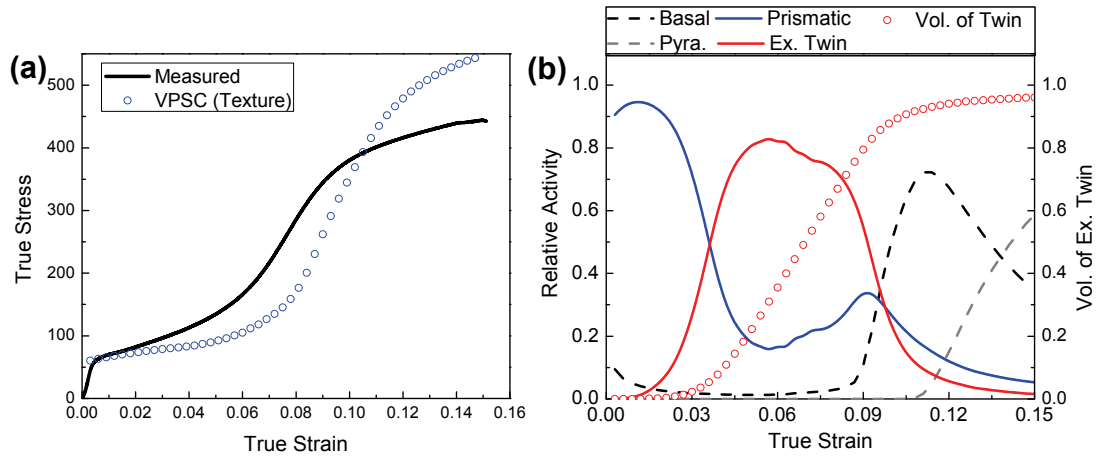


Figure 6-12 The stress-strain behavior (compared with the experimental result) and the relative activities of various deformation modes under compression along TD by using the latent hardening of  $h^{\text{twin,basal}} = 1.2$  ,  $h^{\text{twin,prismatic}} = 1.4$  and  $h^{\text{twin,pyramidal}} = 1$ : (a) stress-strain behavior, and (b) the relative activities of various deformation modes.

Comparing to the texture hardening behavior introduced by the extension twin in the *c*-tension case, the flow stress increases from 120MPa to 350MPa. Therefore, the texture hardening of *a*-compression case is more than 50% stronger than that in the *c*-tension one.

Apparently, if the exact same input parameters as *c*-tension case are used in the *a*-compression case, the stress-strain curve does not fit the experimental results. It was found that the flow stress at the strain from 0.01 to 0.11 obtained from the VPSC simulation is lower than that obtained through the experiment, while that at the strain from  $h^{twin,slip}$  is higher. Therefore, some twin-slip or twin-twin interaction introduced by the microstructure evolution should be considered in addition to the texture hardening effect introduced by extension twinning. Therefore,  $h^{twin,slip}$  of extension twinning on the basal, prismatic, and pyramidal slip can be changed to further improve the VPSC simulated stress-strain behavior under the *a*-compression.

#### 6.5.5.2 Hall-Petch like hardening

According to the relative activities of various deformation modes, the basal and pyramidal slip are the main active deformation mode at the strain from 0.11 to 0.15, Figure 6-12b. Therefore, the decrease in the hardening effect on basal and pyramidal slip is expected to achieve the better fitting of the stress-strain behaviors at the stage. Moreover, it was known that both basal and pyramidal slip is activated in the twinned daughter grain based on the texture evolution analysis and Schmid factor calculation, Figure 6-11b. The latent hardening of extension twinning for basal slip,  $h^{twin,basal}$ , was

decreased from 1.2 to 0.6 and that for the pyramidal slip,  $h^{twin,pyramidal}$ , was decreased from 1 to 0.8 in the  $a$ -compression case to achieve a better fit for the strain range from 0.11 to 0.15 through VPSC simulation, Figure 6-13a. By decreasing the latent hardening of the extension twin on the basal and pyramidal slip, the flow stress decreases from 550MPa to 450MPa at the strain of 0.14. Moreover, the strain where basal slip starts to be activated changes from the 0.09 to 0.07 and the maximum basal slip activity increases from the 0.75 to 0.92 in the daughter grain, since the basal slip is now much easier to be activated within the twins.

It was revealed that the narrow twin bands are formed at the beginning of plastic deformation stage, and the twin thickness increases with the increase in the strain more or less under the  $c$ -tension and  $a$ -compression cases due to the twin growth. The average twin thickness,  $D_t$ , and twin length,  $L_t$ , keep increase as a function of strain under  $c$ -tension and  $a$ -compression cases, Figure 6-6. The thickness of the twin band changes with the increase in the strain and the trends are distinctly different under these two loading paths. According to the microstructure evolution studied through EBSD, it was found that narrow twin band occurs at the beginning of the yielding. The narrow twin band slightly increases with the increase in the strain due to the twin growth. However the twin growth is inhibited by the twin intersection with the other twin band with different variants as a result of the multiple twin variant selection under the  $c$ -tension case.

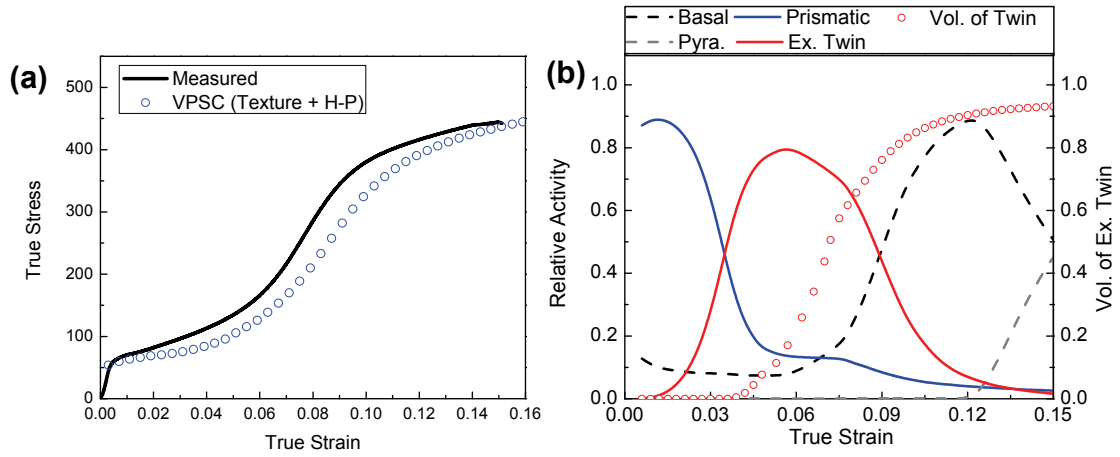
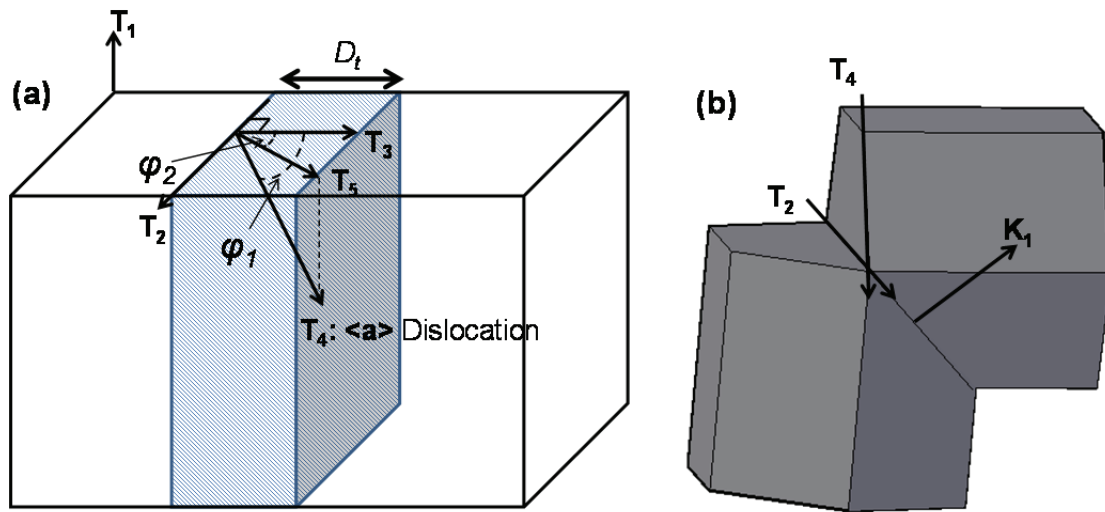


Figure 6-13 The stress-strain behavior (compared with the experimental result) and the relative activities of various deformation modes under compression along TD by using the latent hardening of  $h^{\text{twin,basal}} = 0.6$ ,  $h^{\text{twin,prismatic}} = 1.4$  and  $h^{\text{twin,pyramidal}} = 0.8$ : (a) stress-strain behavior, and (b) the relative activities of various deformation modes.

On the other hand, the narrow twin band shows after the yielding and it keeps growing until it consumes the whole grain to make the whole grain reoriented for the  $a$ -compression case. Therefore, the thickness of the twin band in  $a$ -compression case is much larger than that is observed in the  $c$ -tension case at the large strain stage.

The twin band, which forms as a result of the activation of extension twinning works as the obstacle of the dislocation movement similar to the grain boundaries, and therefore, provide the Hall-Petch (H-P) like hardening effect. The H-P hardening effect introduced by the extension twinning is studied in pure Ti by Kalidindi [41]. The twin boundaries were counted as grain boundaries in the measurement of grain size with the H-P relationship of the Ti to calculate the hardening effect. However, there is no quantitative study on the H-P effect of extension twin for the Mg alloy. Moreover, the lamella shapes of the extension twin in the Mg alloy provide some complications to define the effective grain size or the mean free path.

It was found that the prismatic slip is activated in the daughter grain under the  $c$ -tension case, while the basal slip is the most preferred deformation mode under  $a$ -compression. Therefore, the prismatic slip and basal slip in the daughters under  $c$ -tension and  $a$ -compression should be hardened as a result of the H-P like hardening effect caused by the extension twinning. Also, considering the strong crystal orientation of the parent and the geometry relation between the parent and daughter when specific twin variant is selected, the mean free path (MFP) of the prismatic and basal slips can be calculated based on the geometry relationship as shown in Figure 6-14.



- $T_1$ : the normal of surface  
 $T_2$ : the vector of twin boundary  
 $T_3$ : the vector which is perpendicular to the twin boundary and on the surface plane  
 $T_4$ : the  $\langle a \rangle$  dislocation in the twin  
 $T_5$ : the projection of  $T_4$  on the surface  
 $\varphi_1$ : the angle between  $T_3$  and  $T_4$   
 $\varphi_2$ : the angle between  $T_3$  and  $T_5$   
 $D_t$ : Twin thickness

Figure 6-14 The geometry relation between the twin thickness and mean free path of  $\langle a \rangle$  dislocation: (a) the illustration of geometry relation between the twin thickness and mean free path of  $\langle a \rangle$  dislocation, and (b) the geometry relation of the matrix and twin daughter.

According to the twin thickness,  $D_t$ , and twin length,  $L_t$ , obtained through the EBSD for both  $c$ -tension and  $a$ -compression cases as a function of strain, it was found that the twin length is much bigger than the twin thickness. Therefore, the MFP of the dislocation is dominated by the twin thickness. It should be also noted that the twin width cannot be measured, since only two-dimensional image is provided by the EBSD analysis in the current study. It is assumed that the twin width is similar to the twin length, which is much larger than the twin thickness as well. Therefore, the twin can be assumed as a lamella which can penetrate deep into the matrix, Figure 6-14.

To simplify the calculation, the ideal grain orientation is assumed in the parent, which is  $\{0001\} \langle 10\bar{1}0 \rangle$ . Therefore, the normal of the measurement surface for the  $c$ -tension case is  $\{00.1\}$ , while that for  $a$ -compression case is  $\{10.0\}$ . For the matrix calculation, the crystal vector is converted into the Cartesian coordinate through the transition matrix  $R_C$  as follows:

$$R_C = \begin{pmatrix} 1 & -0.5 & 0 \\ 0 & \sqrt{3}/2 & 0 \\ 0 & 0 & c/a \end{pmatrix} \quad 6-2$$

where  $c/a$  ratio is 1.624 for Mg alloy. Thus, the normal direction of the surface,  $T_1$ , can be expressed as:

$$T_1 = R_C \cdot (0 \ 0 \ 1)^T \quad c - tension \quad 6-3$$

$$T_1 = R_C \cdot (1 \ 0 \ 0)^T \quad a - compression \quad 6-4$$

Since the first undistorted plane  $K_1$  is  $\{1\bar{1}2\}$ , and the difference of the twin variant selection of the two different case,

$$\mathbf{K}_1 = (1 \ -1 \ 2)^T \quad c - tension \quad 6-5$$

$$\mathbf{K}_1 = (1 \ 0 \ 2)^T \quad a - compression \quad 6-6$$

The twin boundary,  $T_2$ , should equals to:

$$\mathbf{T}_2 = \mathbf{T}_1 \times \mathbf{K}_1 \quad 6-7$$

Therefore, the thickness direction,  $T_3$ , on the measured plane should be perpendicular to the normal of the measurement surface and twin boundaries, which can be obtained through:

$$\mathbf{T}_3 = \mathbf{T}_2 \times \mathbf{T}_1 \quad 6-8$$

Assuming both prismatic and basal  $\langle a \rangle$  dislocations in daughter grains under  $c$ -tension and  $a$ -compression, respectively, are both edge dislocation, the slip direction of  $\langle a \rangle$  dislocation,  $T_4$ , is the same as the Burgers vector  $\{10.0\}$ . To meet the self-consistent requirement, the Burgers vector for  $\langle a \rangle$  dislocation in the daughter grain can be  $(1 \ 0 \ 0)_t$  or  $(0 \ 1 \ 0)_t$  for  $c$ -tension, and  $(0 \ 1 \ 0)_t$  or  $(1 \ 1 \ 0)_t$  for  $a$ -compression. Considering the symmetric relationship in the geometry, the angle for each pair should be the same, thus only one for each case is selected for the calculation. Thus,  $T_{4,t}$  can be represented in the Cartesian coordinate in twin system as:

$$\mathbf{T}_{4,t} = \mathbf{R}_c \cdot (1 \ 0 \ 0)^T \quad c - tension \quad 6-9$$

$$\mathbf{T}_{4,t} = \mathbf{R}_c \cdot (0 \ 1 \ 0)^T \quad a - compression \quad 6-10$$

The twin-parent transition matrix,  $R_t$ , is required to transfer the  $T_{4,t}$  into the global (parent) coordinate and normalized as  $T_{4,p}$ . Since the twinning is the  $180^\circ$  rotation of the parent around  $K_1$ . Therefore, the  $R_t$  can be expressed as:



$$\mathbf{R}_t(K_1(n_x \ n_y \ n_z), 180^\circ)$$

$$= \begin{pmatrix} \cos 180 + (1 - \cos 180)n_x^2 & (1 - \cos 180)n_x n_y - (\sin 180)n_z & (1 - \cos 180)n_x n_z + (\sin 180)n_y \\ (1 - \cos 180)n_x n_y + (\sin 180)n_z & \cos 180 + (1 - \cos 180)n_y^2 & (1 - \cos 180)n_y n_z - (\sin 180)n_x \\ (1 - \cos 180)n_x n_z + (\sin 180)n_y & (1 - \cos 180)n_y n_z + (\sin 180)n_x & \cos 180 + (1 - \cos 180)n_z^2 \end{pmatrix} \quad 6-11$$

, and thus,

$$\widehat{\mathbf{T}}_{4,p} = \mathbf{R}_t \cdot \mathbf{T}_{4,t} \quad 6-12$$

It worth mentioning that the twin-parent transition matrix is a special matrix, whose transpose and inverse are both equal to its own.

Therefore, the angle,  $\varphi_1$ , between the  $\mathbf{T}_2$  and  $\widehat{\mathbf{T}}_{4,p}$  can be calculated as:

$$\cos(\varphi_1) = \frac{\mathbf{T}_2 \cdot \widehat{\mathbf{T}}_{4,p}}{|\mathbf{T}_2|} \quad 6-13$$

If we do the projection of  $\widehat{\mathbf{T}}_{4,p}$  on the surface  $\mathbf{T}_1$ , the projected vector  $\widehat{\mathbf{T}}_5$  can be represented as:

$$\widehat{\mathbf{T}}_5(\mathbf{T}_1(n_x \ n_y \ n_z)) = \begin{pmatrix} 1 - n_x^2 & -n_x n_y & -n_x n_z \\ -n_x n_y & 1 - n_y^2 & -n_y n_z \\ -n_x n_z & -n_y n_z & 1 - n_z^2 \end{pmatrix} \cdot \widehat{\mathbf{T}}_{4,p} \quad 6-14$$

Therefore, the angle,  $\varphi_2$ , between the  $\mathbf{T}_2$  and  $\widehat{\mathbf{T}}_5$  can be calculated as:

$$\cos(\varphi_2) = \frac{\mathbf{T}_2 \cdot \widehat{\mathbf{T}}_5}{|\mathbf{T}_2|} \quad 6-15$$

$$MPF = D_t / c \cos(\varphi_1) \text{ or } D_t / c \cos(\varphi_2) \quad 6-16$$

According to the Equation 6-13 and 6-15, the angle,  $\varphi_1$  and  $\varphi_2$  for the prismatic <a> dislocation in *c*-tension and basal <a> slip in *a*-compression can be calculated.

Therefore, the MPF of the prismatic and basal slip in the daughter grains under *c*-tension and *a*-compression as a function of strain can be calculated through the Equation 6-16 based on the evolution of twin thickness,  $D_t$ , and the angle between the slip direction and twin thickness ( $\varphi_1$  and  $\varphi_2$ ).

According to the Hall-Petch relationship study in Chapter 3, the grain size sensitivity of basal and prismatic slip ( $\text{MPa}\cdot\text{mm}^{1/2}$ ) is 2.61 and 5.54, respectively. Therefore, the Hall-Petch like hardening effect introduced by the extension twin can be quantitatively explicated accordingly:

$$\Delta\sigma(\varepsilon) = M(\varepsilon)k_{\tau} \cdot [D^{-\frac{1}{2}} - (MFP(\varepsilon))^{-\frac{1}{2}}] \quad 6-17$$

, where  $D$  is the initial grain size, and  $m(\varepsilon)$  is the Schmid factor at a given strain. Hence, the Hall-Petch hardening effect,  $\Delta\sigma(\varepsilon)$ , as a function of strain can be obtained based on the Schmid factor shown in Figure 6-11 and MPF changes shown in Figure 6-6.

This relationship justifies the reason to lower the extension twinning latent hardening parameter of basal and pyramidal slip in the  $\alpha$ -compression case because the MPF is much larger than that in the  $c$ -tension case. Moreover, the H-P like hardening effect equation can be embedded in the current VPSC simulation by combining the thickness variation of the twin band as a function of strain and the H-P parameters for various deformation modes, , instead of using constant empirical latent hardening parameters.

#### 6.5.5.3 Glissile to sessile hardening

After improving the  $h^{twin,basal}$  and  $h^{twin,pyramidal}$  based on the Hall-Petch hardening, the flow stress in the strain range from 0.11 to 0.15 from the VPSC simulation shows a good match with the experiment data. However, the simulated stress-strain behavior at the strain from 0.01 to 0.11 is still somewhat lower than the experimental results for the  $\alpha$ -compression case. Finally, the latent hardening parameter,

$h^{twin,prismatic}$ , will be improved by increasing it from 1.4 to 1.8 to achieve a better fitting of the stress-strain behavior in this stage, Figure 6-15. The physical justification for this increase in the latent hardening of extension twinning on the prismatic slip in the  $\alpha$ -compression case is that the much lower activity of prismatic slip during the significant activation of extension twin with the increase in the strain has to be modeled via significant increase in the prism hardening within the twins. Basinski [32] reported that dislocations change from the moveable glissile dislocation to the sessile immoveable ones in the daughter grains, which is called as Basinski mechanism. Basinski reported the higher microhardness in the twinned region in face-centered cubic (fcc) metals. However, the interpretation of the observation is still controversial because the twins in the fcc metals are very thin, which is smaller than the microhardness indentations used in the study. Therefore, it is hard to conclude whether the hardening effect came from the Hall-Petch like hardening effect or Basinski effect as they proposed.

Subsequently, Kalidindi [41, 42] extrapolated the stress-strain behavior from the early plasticity stage, where the deformation twinning has not been formed and used the Holloman equation  $\sigma = k\epsilon^n$ . Moreover, the Hall-Petch parameter for Ti is accounted to provide the grain refinement hardening effect caused by the extension twin. The slight difference between curves based on the Holloman equation and the curves with the Hall-Petch relation confirmed the Basinski effect in the Ti. Moreover, the microhardness measurement was performed in the daughter grain as well, which showed higher strengths in the twinned regions.

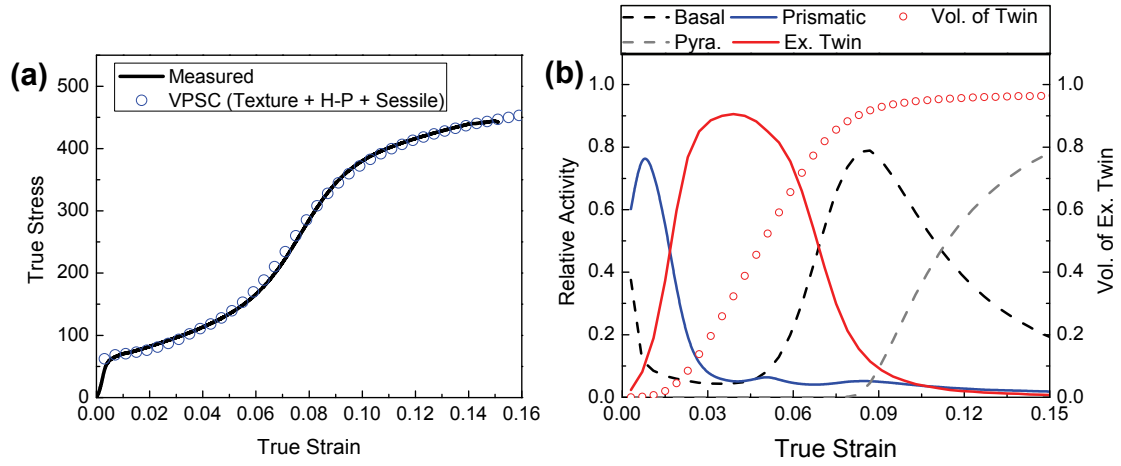


Figure 6-15 The stress-strain behavior (compared with the experimental result) and the relative activities of various deformation modes under compression along TD by using the latent hardening of  $h^{\text{twin,basal}} = 0.6$ ,  $h^{\text{twin,prismatic}} = 1.8$  and  $h^{\text{twin,pyramidal}} = 0.8$ : (a) stress-strain behavior, and (b) the relative activities of various deformation modes.

Meanwhile, little difference in microhardness values measured between the thick twin and thin twin indicated that the hardening effect is related to Basinski effect instead of the H-P hardening.

In the Mg alloy, the VPSC simulation already shows that the prismatic slip occurs earlier than the extension twin in the parent during the  $a$ -compression. Therefore, plenty of prismatic  $\langle a \rangle$  dislocations are activated in the parent. When the extension twin is activated and the twin boundaries pass through in the daughter grain, the prismatic slip is no longer in the suitable geometric relation to the slip. Therefore, the glissile prismatic slip transfers to the sessile one and provides the additional hardening effect. Considering that the extension twin occurs earlier than any slip system in the  $c$ -tension case, the Basinski hardening effect should be much weaker factor. Therefore, the latent hardening parameter  $h^{\text{twin,prismatic}}$  in the  $a$ -compression case needs to be increased to match the stress-strain behavior in the  $a$ -compression.

#### 6.5.5.4 Twin-twin interaction

In this study, the twin-twin interaction was not considered, since little direct evidence to support the hardening effect introduced by the twin-twin interaction was noticed. The twin-twin interaction could have two possible effects: (1) the twin propagation may be inhibited since it is very hard for a twin to penetrate into another twin band and (2) the twin is harder to be activated when the effective grain size becomes very small. In this case, if the twin-twin interaction were considered, the twin-twin interactions in the  $c$ -tension case would be relatively stronger than those in the  $a$ -

compression case, since more than one variant is activated under  $c$ -tension and plenty of twin intersections were observed. However, the clear plateau stage shown in the  $c$ -tension case after the yielding, which indicated that the twin-twin interaction does not occur at the early stage of plastic deformation perhaps because the twins have not propagated or grew large enough to interact within a single grain. From the microstructure evolution measured using the EBSD, the twin intersections may become a factor when the strain reaches about 0.05 for the  $c$ -tension case.

Some papers [16, 20] reported that the difference in the twin nucleation and propagation kinetics between the single variant and multiple variants selection case in terms of the twin volume fraction, twin number, and twin thickness as a function of the applied strain. However, as it is mentioned earlier, the slip activation systems before and after twinning under the  $c$ -tension and  $a$ -compression cases are distinctly different. Thus, the twinning kinetics study based on the applied stress is not very reasonable, since some difference comes from the different strain accommodated by the activation of basal and/or prismatic slip in the matrix and twinned daughter grains. However, it can be suggested that the nucleation mechanism dominates the twinning in the multiple variants selection case, while growth mechanism dominates the twinning in the single variants case.

#### **6.5.6 Overall hardening behavior**

The macroscopic hardening rates as a function of the strain under  $c$ -tension and  $a$ -compression are obtained from the mechanical test and VPSC simulation, Figure 6-16.

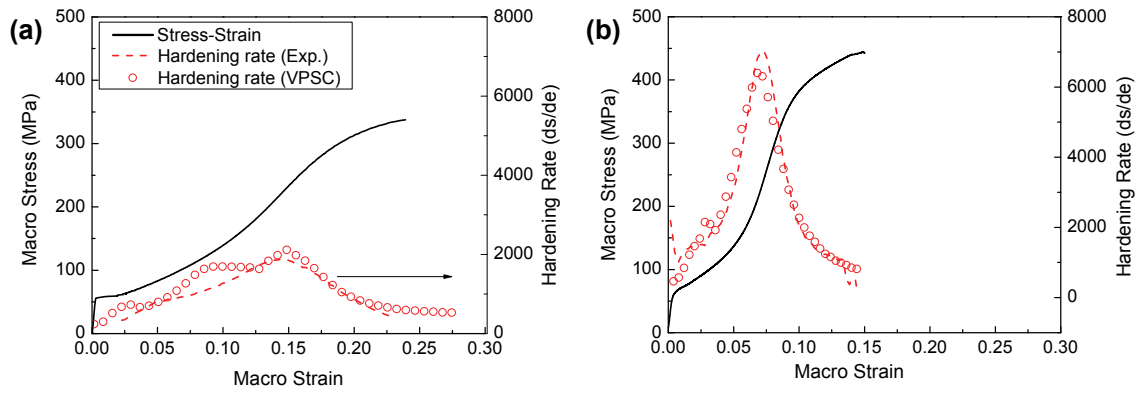


Figure 6-16 The hardening rate, obtained from the mechanical test and VPSC simulation as a function of strain for the tension along TD (*c*-tension) and compression along ND (*a*-compression) loading: (a) tension along TD (*c*-tension), and (b) compression along ND (*a*-compression).



The peak profiles in both cases come from the activation of extension twin. It was found that the hardening rate in the  $a$ -compression case is much higher (6000MPa) than that in the  $c$ -tension case (2000MPa) at the peak, mainly due to the much stronger texture hardening effect in the  $a$ -compression case as discussed earlier. The higher Hall-Petch like hardening effect is achieved in the  $c$ -tension case, while the higher texture hardening and Basinski hardening effects are prevalent during the  $a$ -compression loading.

## 6.6 Conclusion

A commercial hot-rolled Mg AZ31B alloy with strong plane texture where  $c$ -axes of the crystals are almost all parallel to the ND was used to study the effect of extension twin on the microstructure and texture evolution at room temperature using synchrotron x-ray diffraction (S-XRD) and electron backscattering diffraction (EBSD). Two most extension-twin favorable loading paths were investigated: tension along ND (namely  $c$ -tension) and compression along TD (namely  $a$ -compression). Moreover, by using viscoplastic self-consistent (VPSC) modeling, the activation of various deformation modes was studied. The conclusions are as follows.

1. The concave up stress-strain behaviors are observed in both loading paths, which indicates the twin favorable plastic deformation. The yield strengths for  $c$ -tension (56MPa) case is slightly lower than that for the  $a$ -compression case (61MPa). After the yielding, the plateau stage appears under the  $c$ -tension, while there is no obvious plateau stage under  $a$ -compression. Moreover, the hardening of the  $a$ -compression is much

higher than that of *c*-tension with much limited ductility as well. At the strain of 0.15, the flow stress for *a*-compression is 447MPa, while that for *c*-tension is only 232MPa.

2. All three twin variants,  $(10\bar{1}2)[\bar{1}011]$ ,  $(01\bar{1}2)[0\bar{1}11]$ , and  $(\bar{1}102)[1\bar{1}01]$ , are activated under *c*-tension case, since the same Schmid factors (0.499) are achieved for these three variants. However, only two variants  $(10\bar{1}2)[\bar{1}011]$  and  $(\bar{1}102)[1\bar{1}01]$  can be activated under *a*-compression, since their Schmid factor is 0.374, while the Schmid factor for  $(01\bar{1}2)[0\bar{1}11]$  is as low as 0.

3. For the *c*-tension case, the homogenous rim was obtained in the  $\{0002\}$  pole figure, which means that the *c*-axis of the crystal shifted from the ND to the direction parallel to the RD-TD surface with very limited preferred orientation as a result of the extension twinning with multiple variants selection. However, only south/north poles were shown in the  $\{0002\}$  pole figure after *a*-compression loading. Therefore, *c*-axis of the crystal shifted from the ND to the TD with very strong preferred orientation as a result of the twinning with only one variant selected in most of the matrix.

4. The narrow twin bands with plenty of twin intersections were formed after the *c*-tension deformation since multiple twin variants were selected. The effective grain size is much smaller than the initial grain size and the twin boundaries caused significant Hall-Petch like hardening. In contrast, narrow but parallel twin bands with a single variant formed at the beginning of the plastic deformation during *a*-compression loading. The twin thickness increased during the deformation until the twins came together with the neighboring twins and formed larger combined twins occupying the entire matrix. It resulted in a complete reorientation of almost entire parent grains when the strain was

about 0.08. Therefore, the effective grain size was similar to the initial grain size at this case.

5. The extension twin is activated first and followed by the prismatic slip in the twinned daughter grains in the *c*-tension case, while prismatic slip is activated earlier than the extension twinning under *a*-compression case. Although both basal and prismatic slip have very limited Schmid factor, basal slip is activated to accommodate the strain in the daughter grain in the *a*-compression case because of its relatively lower critical resolved shear stress and followed by the activation of pyramidal slip at the high stress.

6. The prismatic slip is in the favorable orientation in the twinned daughter grain under *c*-tension, while both basal and prismatic slips are in the hard orientation in the daughter grain under *a*-compression case. Therefore, the much higher texture hardening effect is obtained during the *a*-compression loading, which increases the stress from 100MPa at the strain of 0.04 to 375MPa at the strain of 0.10.

7. The much higher Hall-Petch like hardening effect is obtained during the *c*-tension case, because of the much smaller effective grain size after the activation of twinning. The narrow twin bands with various twin variants intersect with each other to hinder the twin growth so that the effective grain size becomes much smaller than the initial grain size. On the other hand, the narrow twin band keeps growing until it makes the whole grain reoriented for the *a*-compression loading. Thus, the effective grain size is similar to the initial grain size. The difference in the mean free path of the prismatic  $\langle a \rangle$  dislocation under *c*-tension and basal  $\langle a \rangle$  dislocation causes the different Hall-Petch like hardening effect, which can be used to explain the requirement of the decrease in the latent

hardening parameters,  $h^{\text{twin,basal}}$  and  $h^{\text{twin,pyramidal}}$ , in the VPSC simulation of the  $a$ -compression to achieve the better fitting to the experimental data.

8. The higher Basinski hardening effect is obtained through the  $a$ -compression case, where the prismatic slip occurs earlier than the extension twin, which can explain the increase in the latent hardening parameters,  $h^{\text{twin,prismatic}}$ , in the VPSC simulation of the  $a$ -compression.

## Chapter 7

### Summary and Conclusions

A commercial hot-rolled Mg AZ31B alloy with strong plane texture where  $c$ -axes of the crystals are almost all parallel to the normal direction (ND) was used to study the effect of grain size and texture on the anisotropic plasticity including the yielding and hardening behaviors at ambient temperature. Various grain sizes were obtained through the isochronal annealing from 523K to 723K, while the systematical changes in the texture were generated by changing the loading orientations,  $\theta$ , with respect to the ND under uniaxial tension and compression. The microstructure and texture evolution during the plastic deformation were investigated by using synchrotron x-ray diffraction (S-XRD) and electron back-scatter diffraction (EBSD) measurements. The activation of various deformation modes including the basal, prismatic, and pyramidal slip as well as extension twin were investigated using the visco-plastic self-consistent (VPSC) model simulation based on the Voce hardening law and predominant twin reorientation (PTR) scheme.

First, the effect of the initial texture on the H-P relationship of the Mg alloy was investigated by changing the loading orientation,  $\theta$ , from 0°-90°. The H-P parameters of friction stress,  $\sigma_0$ , and grain size sensitivity,  $k_\sigma$ , vary significantly depending on the loading direction, since the crystal orientation factor and dominant deformation mechanisms at the yielding stage change with the changes in the loading orientation. Moreover, the H-P relationship for various deformation modes was established based on

the dislocation pile-up theory. The grain size sensitivity  $k_{\tau}^{\beta}$  (MPa·mm<sup>1/2</sup>) increases from basal (2.61), prismatic (5.54), to pyramidal (9.86). On the other hand, extension twinning has higher  $k_{\tau}$  (3.48) than the basal slip (2.61), while it is lower than prismatic slip.

Based on the results from the H-P relationship study, the critical resolved shear stress,  $\tau_o^{\beta}$ , for each deformation mode,  $\beta$ , was obtained and used as the input crystal constitutive parameters in the subsequent VPSC simulation. Other hardening parameters were iteratively optimized by comparing the stress-strain behaviors and texture evolutions under various loading paths and loading orientations to the experimental results. A single set of input crystal constitutive parameters successfully simulated all five tensile and three compressive orientations. Various deformation mechanisms dominate the plastic deformation for the specimens with different initial textures. The extension twin, which provides significant texture evolution, is activated first during the plastic deformation at the tension along 0° and 22.5°. Since the crystal reorients to 86.3° to the loading axis during the twinning, the dominant deformation mechanisms change to the prismatic slip in the twinned daughter grains in these loading orientations. The basal slip is activated first during tension along 45° and 67.5°. Some extension twin is still active during tension along 45° due to the angular deviation in the grain orientation in the initial texture. Prismatic slip dominates the plastic deformation during tension along 90° followed by the pyramidal slip. The kinetics of extension twin with the applied strain decreases with the increase in the loading angle during the uniaxial tension. On the other hand, during compression along 0°, the basal slip is first activated but with very limited activities followed by the pyramidal slip. The basal slip is the dominant deformation

mode for the 45° case. During the compression along 90°, the prismatic slip is activated first at yielding stage followed by the extension twin. The basal slip is the dominant deformation mode followed by the pyramidal slip within the twinned daughter grains. The current VPSC study provides a detailed micromechanical understanding of the slip/twin activities and their influence on the hardening behavior.

The VPSC simulation was further improved by incorporating the H-P relationships of various deformation modes in the current study. It is demonstrated that the VSPC simulation can predict the stress-strain behavior and texture evolution for various tensile loading orientations and grain sizes. The H-P parameters obtained from the dislocation pile-up theory were also compared to the results of the H-P VPSC simulation.

The plastic anisotropy dominated by the extension twin was investigated through the comparison of two favorable twin loading paths: tension along ND and compression along TD of the rolled Mg plate. Three twin variants are selected in the tension along ND case, which forms narrow twin bands with 60° intersections, which reorient the {0002} poles from the ND to along TD-RD plane homogenously. On the other hand, the single twin variant is selected in the compression along TD case, which forms parallel twin bands that cause the entire grain to reorient throughout the specimen at the strain of about 0.08. The {0002} component reorients from ND to RD. The significant changes in the hardening behaviors introduced by the extension twin were also investigated in these two loading paths. A significant H-P like hardening effect was introduced in the tension along

ND case, while higher texture hardening and Basinski hardening were observed in the compression along TD case.



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