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The Role of Twinning in the Initiation of Fracture in Am30 and Az61 Magnesium Alloys

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THE ROLE OF TWINNING IN THE INITIATION OF FRACTURE IN AM30 AND AZ61 MAGNESIUM ALLOYS

By

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THE ROLE OF TWINNING IN THE INITIATION OF FRACTURE IN AM30 AND AZ61 MAGNESIUM ALLOYS

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Magnesium alloys are excellent material candidate to reduce mass of automotive structures, and as such to meet the Department of Energy's targets in fuel economy and clean energy. However, magnesium alloys show poor ductility at room temperature, which is one of the most important impediments to achieving cost-effective manufacturing of wrought alloys and insuring good energy absorption in crash structures. This Master thesis aims to identify the mechanisms behind the low ductility of magnesium. Therefore, non-destructive EBSD analyses upon tension of both a strong and weak textured magnesium alloy were conducted with a focus on the role of twinning in fracture initiation. This study revealed five mechanisms responsible for early fracture, all of which relate to twinning activity. These mechanisms were involved directly in the shear incompatibility arising from interactions between twin-twin, twin-slip, twin-grain boundary, and double twinning. Backstress played a major role in twin-grain boundary and twin-twin boundary interactions.

Keywords: Interrupted EBSD, magnesium, twin nucleation, twin propagation, twin-twin hardening

DEDICATION

I would like to dedicate this research to my mother who has always supported me in every aspect of my life.

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TABLE OF CONTENTS

Page

DEDICATION ii

ACKNOWLEDGEMENTS iii

LIST OF TABLES v

LIST OF FIGURES vi

CHAPTER

I. INTRODUCTION 1

II. EXPERIMENTAL PROCEDURE 4

2.1 Materials and Characterization Procedure 4
2.2 Initial State 5
2.3 Mechanical Testing 10
2.4 Fractography 12

III. RESULTS AND DISCUSSION 14

3.1 Slip-Twin 14
3.2 Twin-Twin 16
3.3 Twin-Grain Boundary 17
3.4 Backstress 19
3.5 Double Twinning/Contraction Twin 21

IV. CONCLUSIONS 25

REFERENCES 26

LIST OF TABLES

TABLE Page

2.1 Elemental Composition of Magnesium Alloys 4

LIST OF FIGURES

FIGURE Page

2.1 Optical Micrographs with image analyses of the AM30 alloy at (a) the initial state, 0%, and (b) after 12% of true strain. It was concluded from these optical analysis results that the area fraction particles remained constant from during tensile testing, with almost no voids nucleating on them. 6

2.2 (a) Inverse pole figure maps (IPF) maps obtained by *in situ* EBSD of an AM30 alloy deformed in tension along the extrusion direction, ED, giving nominal twinning conditions (b) IPF maps obtained by *in situ* EBSD of an AM30 alloy deformed in tension along the transverse, TD, giving profuse twinning conditions. 7

2.3 (a) Inverse pole figure maps (IPF) maps obtained by *in situ* EBSD of an AZ61 alloy deformed in tension along the extrusion direction, ED, giving nominal twinning conditions (b) IPF maps obtained by i*n situ* EBSD of an AM30 alloy deformed in tension along the TD under profuse twinning conditions. 9

2.4 Stress-strain behavior of (a) AM30 (a) and (b) AZ61 Magnesium Alloys upon tension both extrusion (ED) and transverse (TD) direction at room temperature and a strain rate of 10^{-3} s^{-1}. AM30 shows a higher anisotropy than AZ61 because of the sharper texture which gave more profuse twinning upon tension along the TD. 11

2.5 Fracture Surface of an AM30 sample tested in tension along the extrusion direction which induced nominal twinning conditions and deformation dominated by slip. The fracture surface shows substantial cleavage-like terraces separated by very tourmented regions. Higher magnfication showed that these tourmented regions corresponded to complex agglomerations of dislocation structures 12

2.6 Fracture Surface of an AM30 sample tested in tension along the transverse direction which induced profuse twinning conditions and deformation dominated by twinning. The fracture surface shows substantial cleavage-like terraces. 13

3.1 Twin-Slip Interaction Resulting in Crack Nucleation 15

3.2 Twin-Twin Interaction Resulting in Crack Nucleation 16

3.3 Twin-Grain Boundary Interaction Resulting in Crack Nucleation 19

3.4 IPF maps obtained *In situ* EBSD of an AM30 alloy deformed in tension under nominal twinning conditions showing (a) initial state, (b) after 2% plastic strain with backstress-induced two twins nucleating at the GB separating two grains of very low SF as illustrated in –d, (c) after 4% plastic strain with backstress-induced twinning at the triple point, and (d) pole figure illustrating the orientation of the c-axis of the parent (blue), twins (magenta), and (orange) twins with respect to the loading direction (LD). 20

3.5 (a-b) SEM micrographs showing crack nucleation along two double twin variants exactly following follows the zig-zag meandering of the twins. Observe that the opening of the crack is preferentially located at the intersection of the two twin variants. (c) an IPF from an EBSD scan of the region highlighted in –a that couldn‘t resolve the narrow twins due to the high kinking generated at the free surface, and (d) is image quality map exhibiting better the twins. 22

3.6 (a) an SEM micrographs showing a transgranular crack nucleating at the intersection between a tripe point and needles of twins, and (b) an IPF from an EBSD scan of the region highlighted in –a that resolved the needle as a twin inducing approximately 56° of the blue parent. 23

3.7 *In situ* EBSD orientation maps of an AM30 deformed in tension under profuse twinning conditions showing texture evolution from (a) the initial state, through (b) 2% plastic deformation, and up to (c) 6% plastic deformation with (d) a misorientation profile map across the twins marked by arrows. Massive i.e. double twinning (arrows) preferentially accompanying twins indicating a strong non-Schmid‘s effects. The double twins have the dark blue orientation..................24

CHAPTER I

INTRODUCTION

The reduction of greenhouse gases (GHG) and reduced dependence on hydrocarbon-based fuels is a key priority worldwide. The United States, Canada, China and the Euro-zone have engaged, in a first stage plan to reduce the mass of CO_2 emitted by passenger vehicles below 95g/km (for EU and China) and 110g/km (for the USA and Canada) by 2020. These demands correspond to reductions of GHG emission ranging from 30 – 50% with respect to current standards. Such tremendous improvements will require extensive vehicle mass reduction through the use of lightweight materials allowing for [1] a net fuel economy improvement in combustion engine and [2] an increase in the range of electric vehicles. In addition, all material developments must also maintain/improve crash worthiness of current vehicles. Many metals which may satisfy these conflicting demands exhibit either a hexagonal close-packed (HCP) crystal structure and/or twinning or martensitic transformation-induced plasticity. Candidate materials systems are based upon Mg, Ti, as well as third-generation advanced-high strength steels (AHSS), including twinning-induced plasticity (TWIP), and transformation-induced plasticity (TRIP) steels.

Current structural applications of HCP Mg alloys, in particular, are limited to castings, due to difficulties associated with forming wrought alloys. Increased application of these advanced lightweight materials within the transportation sector will require improvement in both the formability and the ability to absorb energy during crash. It is widely understood that the present limitations in these properties are associated with the plastic anisotropy of the HCP crystals. The dislocations which accommodate plasticity in HCP crystals generally have Burgers vectors within the basal plane. Thus, these materials suffer from an inability to easily deform along the HCP crystal c-axes, in the absence of a deformation twinning-based mechanism of strain accommodation. Deformation twinning has long been known to alleviate the requirement to satisfy the von Mises criterion for polycrystalline plasticity that demands five independent easy dislocation slip modes [1]. However, deformation twinning-based plasticity can be a ―double-edged sword." There have been countless examples in the scientific literature, where twinning is associated with fracture initiation and there are a number of micro-mechanism hypotheses to explain these phenomena. Some of these hypotheses relate to the effect of:

1. Dislocation accommodation of twinning;
2. Additional hardening due to *transmutation* of dislocations at the twin interface;
3. Twin-twin interactions, including twin transmutation and double twinning;
4. Backstress generation due to interactions between twins and with grain boundaries (GBs);
5. Damage initiation associated with twinning (including that due to plastic strain localization within twins and that due to incompatibilities at grain boundaries, GBs, and triple lines.)

The goal of the proposed work is to identify the mechanisms that drive fracture initiation in Mg alloys. We have chosen to use non-destructive EBSD analyses upon tension in an effort to be able to draw conclusive results on the role of twinning on damage. This presents an unprecedented method in literature and as such a unique contribution of this Master work. The challenge, however, was substantial because a same region must be scanned and analyzed by EBSD techniques each time a test up to a small strain level is performed without scratching the sample or destroying by the testing and handling procedures.

Understanding of the aforementioned mechanisms will greatly aid engineering efforts to render Mg "formable" and "crushable," so that society can exploit the performance and efficiency benefits of lightweight Mg alloys. The effects of alloying (both solid solution and precipitation) on individual dislocation and twin-based deformation mechanisms are only beginning to be understood. Without a detailed understanding of these interactions, alloy and microstructure design efforts will proceed in an empirical, data-driven manner at a pace too slow for incorporation into modern engineering applications.

CHAPTER II

EXPERIMENTAL PROCEDURE

2.1 Materials and Characterization Procedure

In this study, two magnesium alloys, namely extruded AM30 and AZ61, were used. The extrusion was performed in plane strain condition to promote fiber texture instead of a more complex rod texture. The plane strain condition of shaping was achieved by extruding a hollow crash rail which had flat sections.

The motivation behind using these two alloys is to probe damage in a highly anisotropic material in comparison to that in a material with less anisotropy. It was then necessary to use two different compositions. The composition of AZ61 is such that it will generate relatively weaker texture than that obtained by extruding AM30. The reason lies on the effect of solutes, particles and cross slip activities on dynamic recrystallization.

Elemental spectroscopy was conducted on both crash rails and the results are depicted in Table 2.1.

Table 2.1 Elemental Composition of Magnesium Alloys

Alloy	**Mg**	**Al**	**Mn**	**Zn**	**Si**	**Fe**	**Ni**	**Cu**
AM30	Bal	2.83	0.368	0.0037	0.0083	0.0061	<0.0015	<0.0005
AZ61	Bal	5.94	0.277	0.73	0.025	0.012	<0.0015	<0.0005

Interrupted electron backscattered diffraction (EBSD) was conducted in order to quantify twinning in AM30 and AZ61 magnesium alloys and identify how damage is related to slip and twinning. The EBSD scans were performed at a step size of 0.1 μm using a TSL EDAX detector within a Zeiss Supra field emission scanning electron microscope (FEG-SEM) at the Center for Advanced vehicular systems (CAVS). EBSD analyses were performed using the commercially available TSL software. Optical microscopy (OM) was performed using a Zeiss Axiovert inverted microscope with image analyses software techniques.

2.2 Initial State

Figure 2.1 shows OM micrographs of both AM30 before deformation. Figure 2.2 depicts inverse pole figure maps of the initial state of the two regions followed by EBSD and FEG-SEM for AM30 in the extrusion direction (ED) and transverse direction (TD), respectively. Figure 2.3 figure maps of the initial state of the two regions followed by EBSD and FEG-SEM for AZ61 in the extrusion direction (ED) and transverse direction (TD), respectively.

Figure 2.1 Optical Micrographs with image analyses of the AM30 alloy at (a) the initial state, 0%, and (b) after 12% of true strain. It was concluded from these optical analysis results that the area fraction particles remained constant from during tensile testing, with almost no voids nucleating on them.

Interrupted electron back scatter diffraction (EBSD) analyses of a same region were conducted on four specimens to quantify twinning. Specimens from AM30 and AZ61 alloys in both the extrusion and transverse direction were chosen in order to quantify the twin in nominal and profuse conditions. The specimens were polished using various grits of silicon carbide paper in order to remove the outer skin of finer grains in order to reveal the larger and more indicative grains. After approximately 0.25mm of material had been removed and the specimen has been polished using 4000 grit silicon carbide paper, the specimens were electrochemically polished.

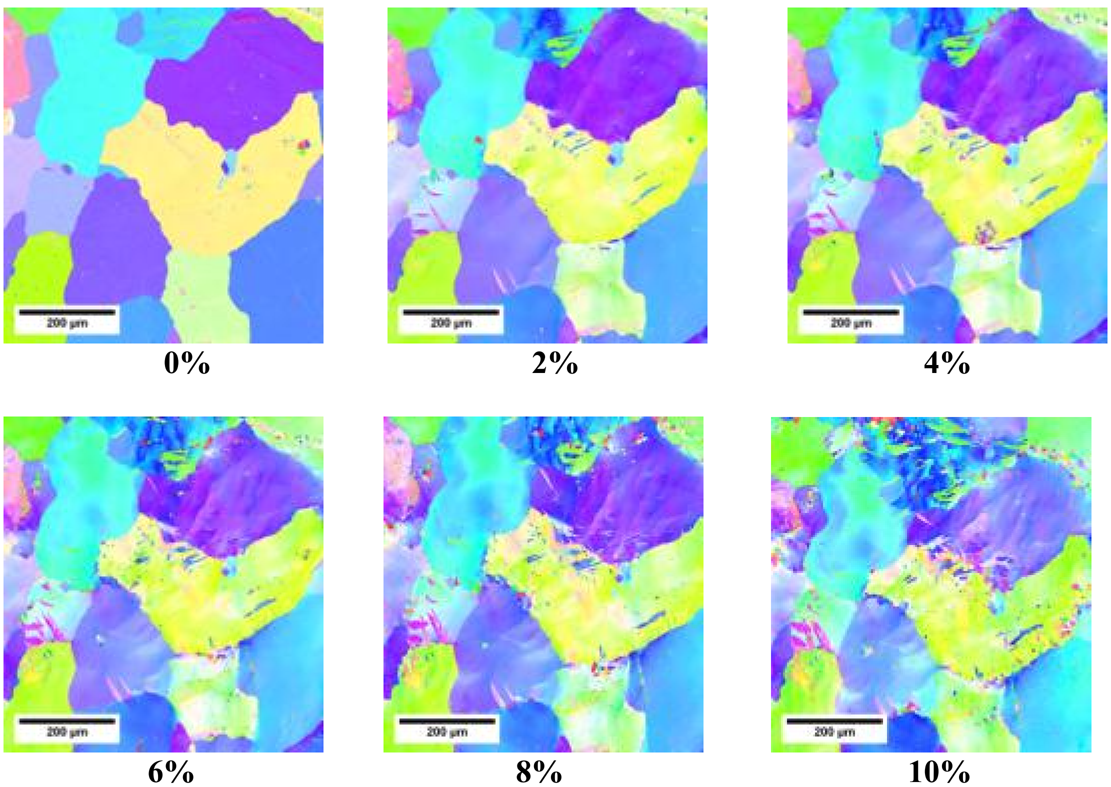

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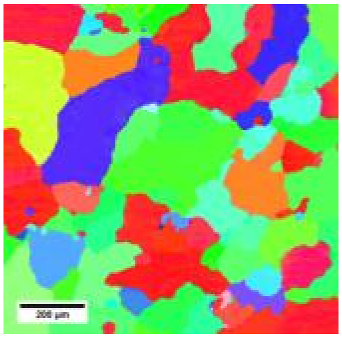
Figure 2.2 (a) Inverse pole figure maps (IPF) maps obtained by *in situ* EBSD of an AM30 alloy deformed in tension along the extrusion direction, ED, giving nominal twinning conditions (b) IPF maps obtained by *in situ* EBSD of an AM30 alloy deformed in tension along the transverse, TD, giving profuse twinning conditions.



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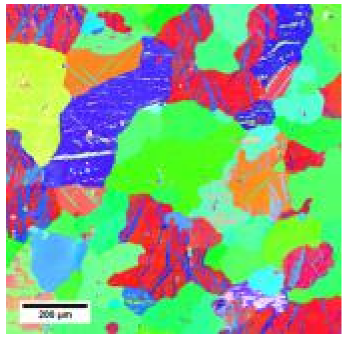

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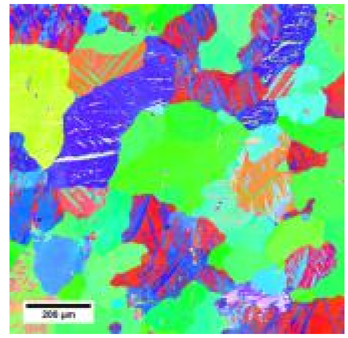

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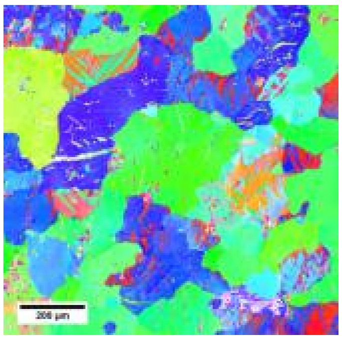

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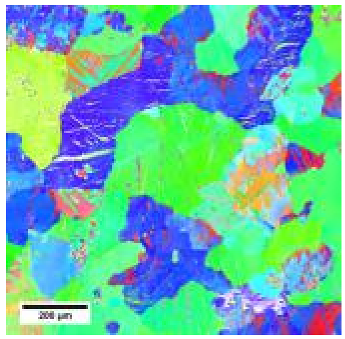

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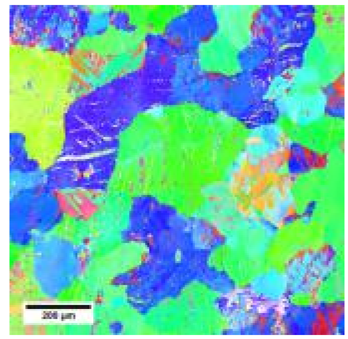

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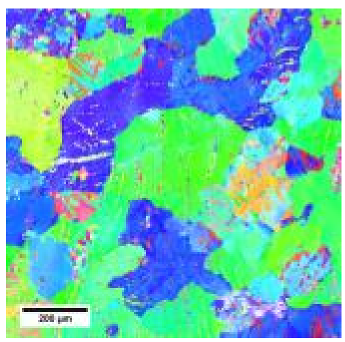

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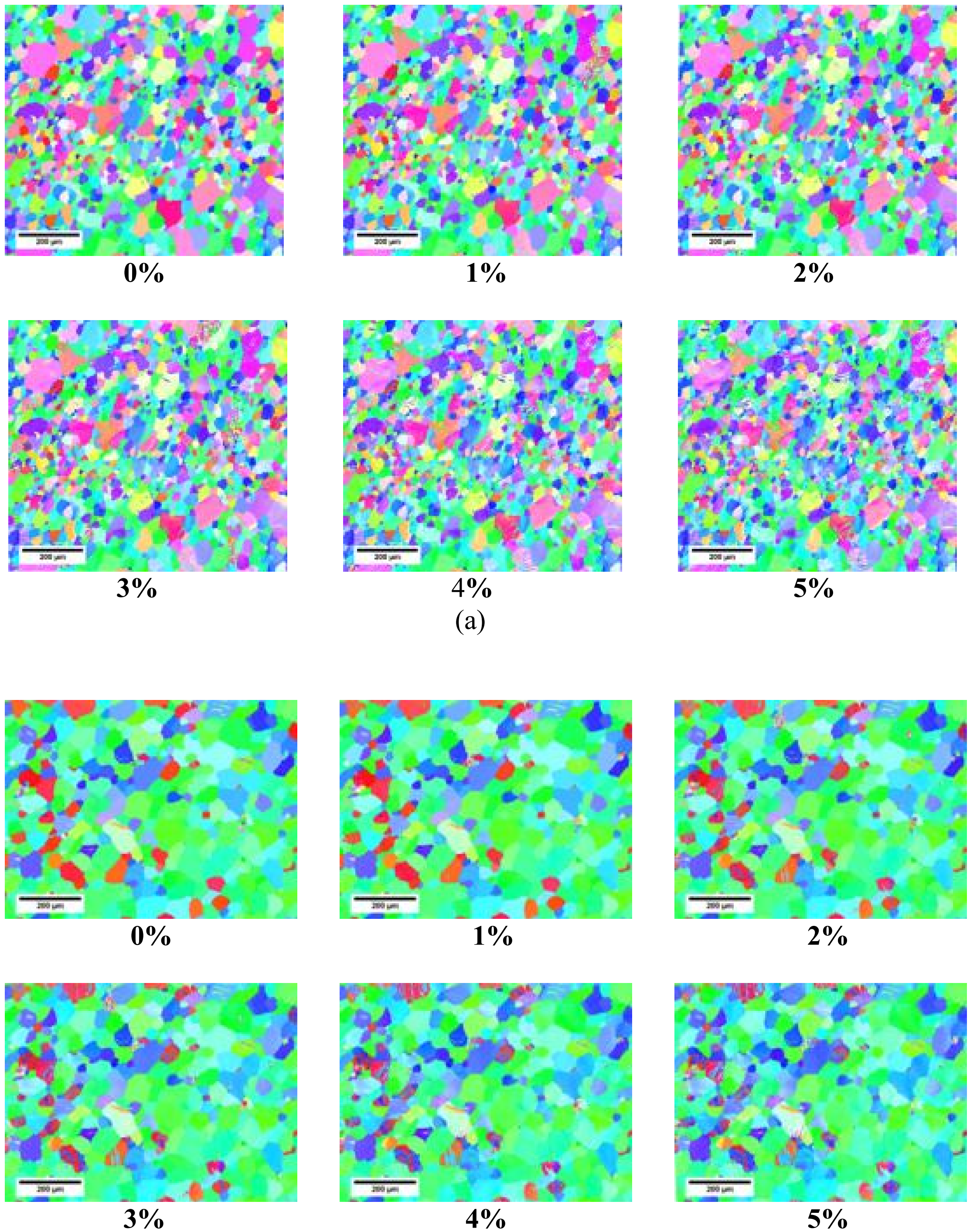
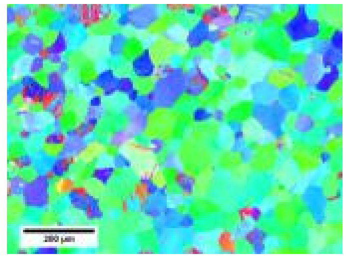


Figure 2.3 (a) Inverse pole figure maps (IPF) maps obtained by *in situ* EBSD of an AZ61 alloy deformed in tension along the extrusion direction, ED, giving nominal twinning conditions (b) IPF maps obtained by i*n situ* EBSD of an AM30 alloy deformed in tension along the TD under profuse twinning conditions.



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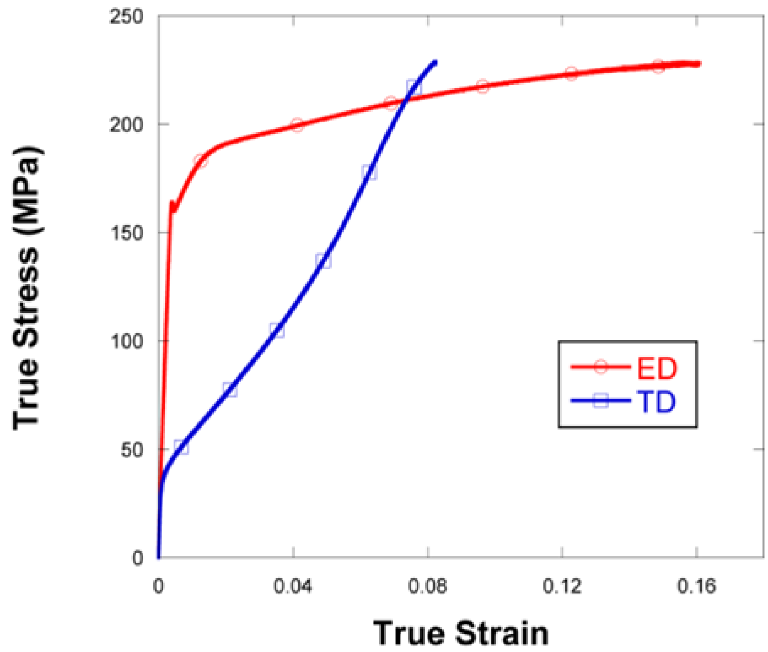
2.3 Mechanical Testing

The specimens were machined from the hollow crash rail in both the extrusion and transverse directions. Monotonic quasi-static tension testing was then conducted at 0.001/s strain rate using an Instron 8856 to determine the mechanical response. The results of the mechanical testing were shown in Figure 2.4 for both AM30 and AZ61 under both loading directions. AM30 exhibited a higher anisotropy than AZ61 because, in particular, the profuse twinning conditions (TD tension) induced the stress-strain behavior to inflect in AM30 but not in AZ61.



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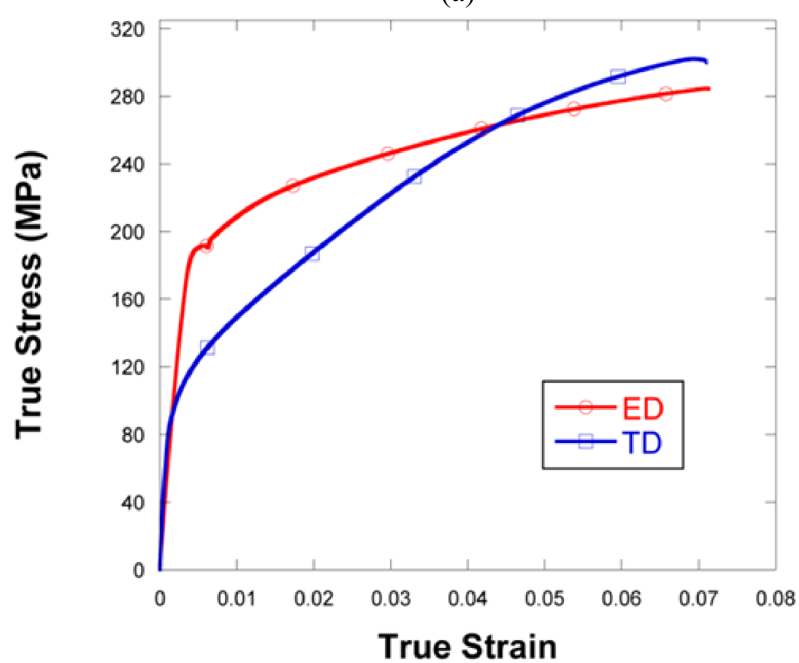

(b)

Figure 2.4 Stress-strain behavior of (a) AM30 (a) and (b) AZ61 Magnesium Alloys upon tension both extrusion (ED) and transverse (TD) direction at room temperature and a strain rate of 10^{-3} s^{-1}. AM30 shows a higher anisotropy than AZ61 because of the sharper texture which gave more profuse twinning upon tension along the TD.

2.4 Fractography

In order to reduce oxidation of the fracture surface, the specimen were placed in the scanning electron microscope (SEM) for immediate imaging of the fracture surface after the tension test had been conducted. The results of FEG-SEM fractography for AM30 under tension along ED and TD are reported in Figures 2.5 and 2.6, respectively.

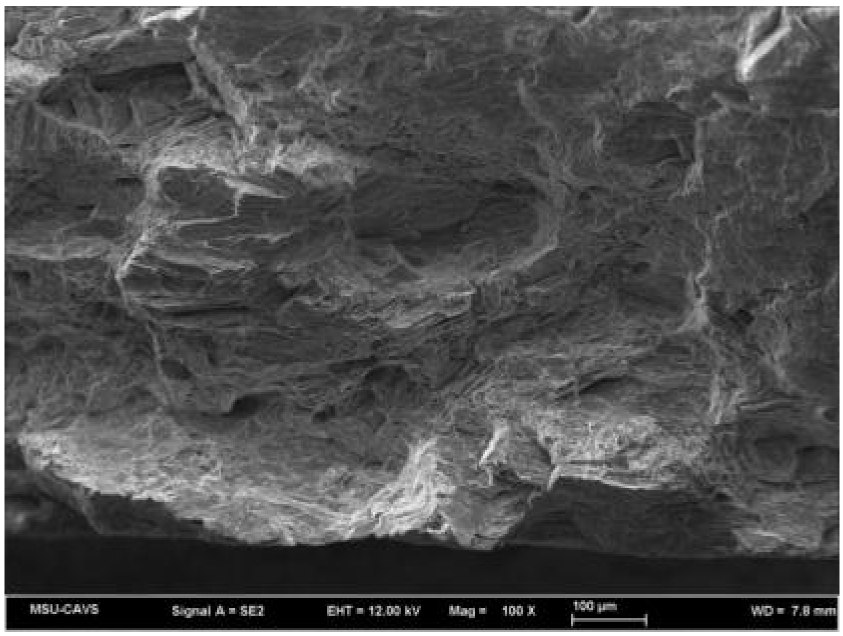

Figure 2.5 Fracture Surface of an AM30 sample tested in tension along the extrusion direction which induced nominal twinning conditions and deformation dominated by slip. The fracture surface shows substantial cleavage-like terraces separated by very tourmented regions. Higher magnfication showed that these tourmented regions corresponded to complex agglomerations of dislocation structures

The fracture surface of AM30 strained in the extrusion direction is shown in figure 2.2. The micrograph of the fracture surface shows mixed modes of fractures. There are flat, cleavage like facets surrounded by more tormented regions.

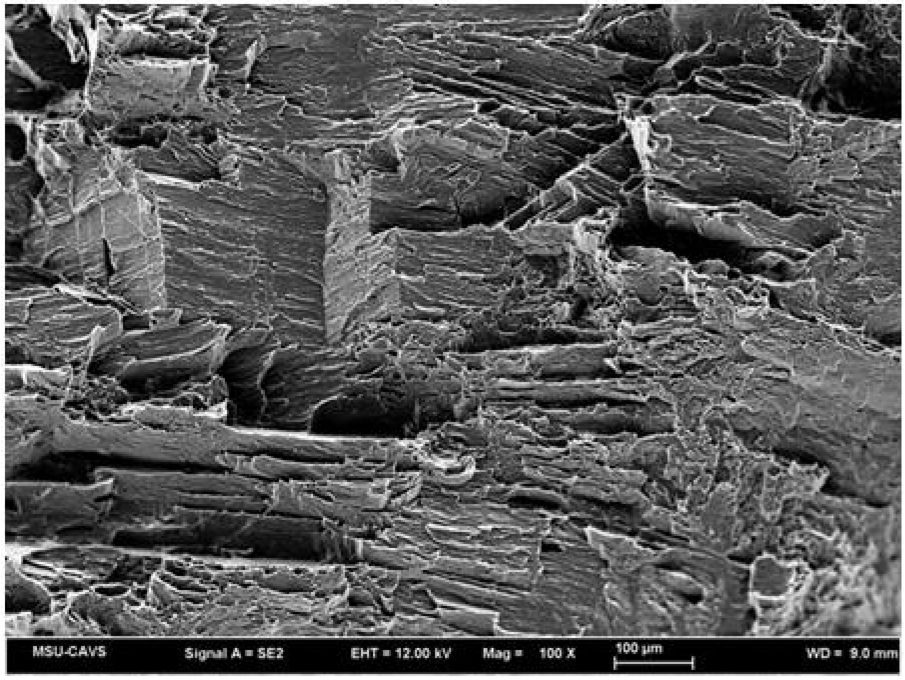

Figure 2.6 Fracture Surface of an AM30 sample tested in tension along the transverse direction which induced profuse twinning conditions and deformation dominated by twinning. The fracture surface shows substantial cleavage-like terraces.

The micrograph of the fracture surface shows more of a single mode failure when compared to the fracture surface of the extrusion direction. The fracture surface is almost exclusively flat, cleavage like facets. The tent shaped facets indicate the specimen fractured along twin boundaries.

CHAPTER III

RESULTS AND DISCUSSION

As a result of the experiments conducted, five mechanisms were found to effect damage from twinning. Slip-Twin, Twin-Twin, Twin-Grain Boundary, Backstress, and Double Twinning.

3.1 Slip-Twin

We invoke here the most important class of slip-twin interactions; which is dislocation transmutation by the twinning interface. Upon incorporation in the twins, the parent dislocations transmute to high order sessile dislocations and/or to other types of glissile dislocations not generated in the twins according to Schmid's law [3–6]. When a parent dislocation encounters a moving twin boundary under the action of stress, the dislocation is engulfed by the twin and leaves a disconnection at the twin interface, thereby releasing another type of dislocation in the twin. It has been demonstrated [7–9] that transmutation is the principal cause of Regime II, which characterizes the sigmoidal stress-strain curve under profuse twinning. These authors argued that the glissile and sessile dislocations generated by transmutation inside the twin are quite different from the slip-dislocations needed to accommodate deformation within the twins. Interactions between Schmid's dislocations under the applied stress and the transmuted dislocations upon twin propagation lead to an abnormal latent hardening in the twins. This latent hardening must be of increasing rate since more dislocations are transmuted by the twins upon further growth of the twins and further parent deformation. The increasing rate of this latent hardening is attuned with the observed increasing hardening rate of Regime II.

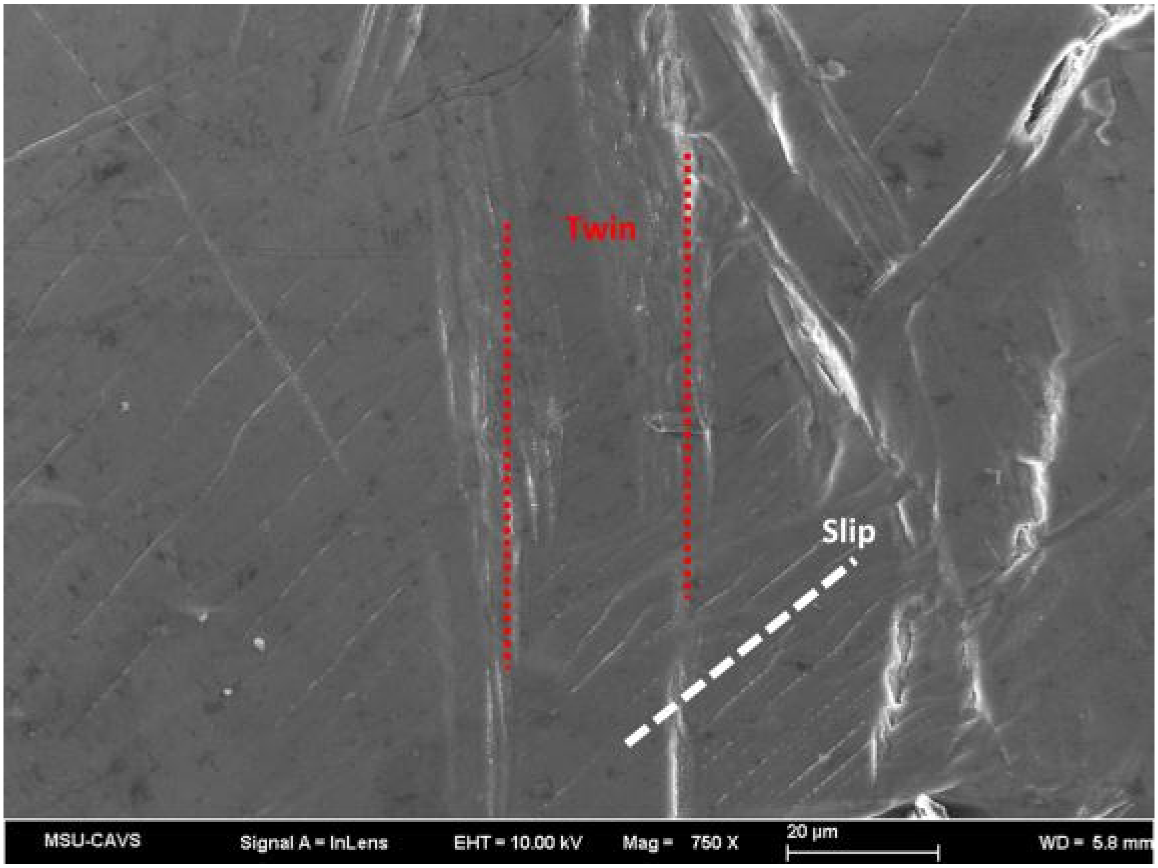

Figure 3.1 Twin-Slip Interaction Resulting in Crack Nucleation

Transmutation has been suggested to drive many other mechanisms related to nucleation of compression twins within the tensile twins, crack nucleation within the twins, and dynamic recrystallization observed at ambient temperature in Mg [10], [11]. Although transmutation has been observed to depend on the reaction at the twin interface, it is still assumed by crystallographers to follow a pure geometrical law based on the correspondence matrix rule (CMR).

3.2 Twin-Twin

Although twin-twin interactions [12–16] appear to have a considerable effect on hardening and fracture, they have received even less attention than slip-twin interactions. Twin-twin and slip-twin interactions are similar in that a stress concentration and a reaction are needed to incorporate twinning dislocations within an obstacle twin. However, the difficulty in modeling twin-twin interactions lies in the interfacial nature of the twinning dislocations to be incorporated. For the shear of an incident twin to be accommodated by the obstacle twin, four options are possible : 1) retwinning of the obstacle twin (here dubbed ―autotwinning"), 2) slip in the incident twin, 3) slip in the obstacle twin, and 4) detwinning of the incident twin [13], [17], [18].

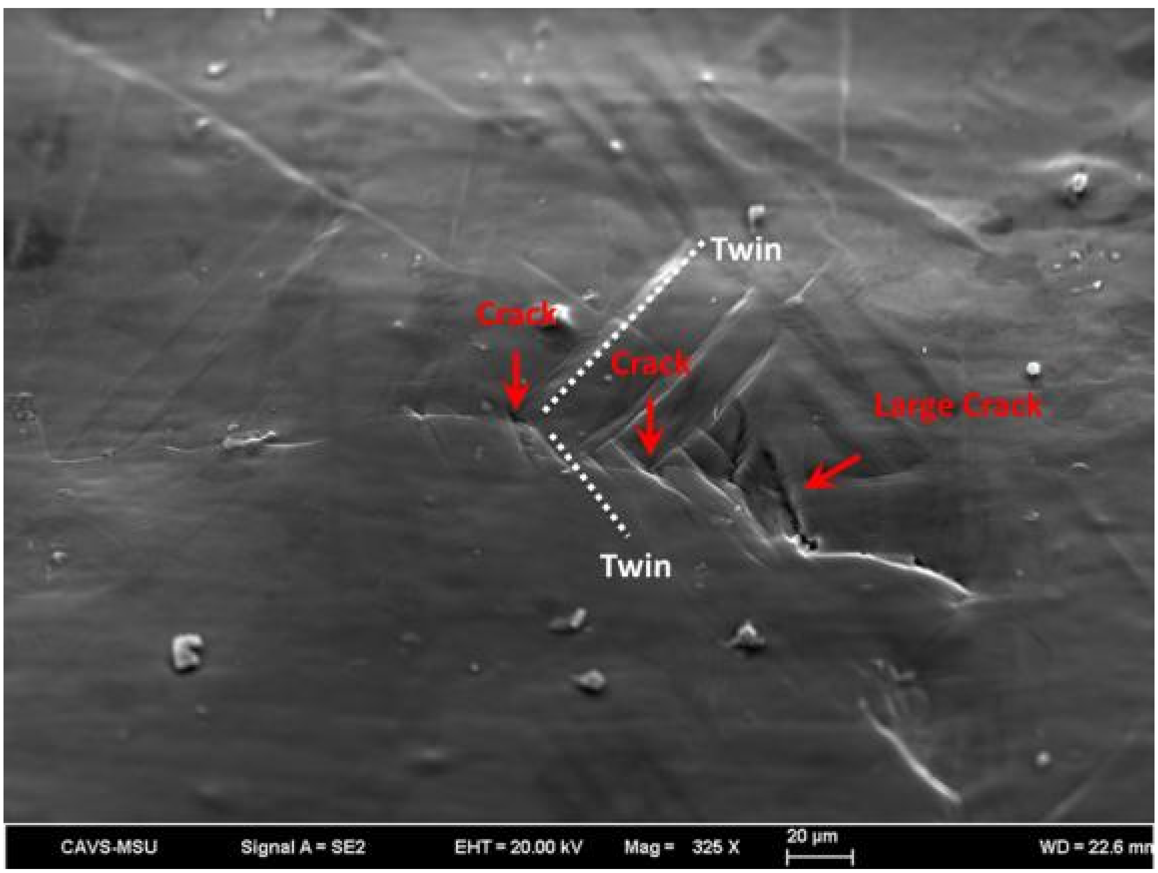

Figure 3.2 Twin-Twin Interaction Resulting in Crack Nucleation

As the critical stresses for nucleation are higher than those for propagation [19], the grain with multivariant twinning is likely to strain harden more rapidly. The effect of twin-twin interactions on fracture was invoked as early as 1868 by Rose as reported by Reed-Hill [20–22] and confirmed by Saito [23] for FCC copper and by Sleeswyk [24], [25] for BCC iron. Recently, MD simulations of Mg under tension showed that cracks readily nucleate at the intersection between twins. Rémy [16] suggested that twin boundaries are subject to more severe compatibility requirements than GBs, at least in FCC crystals, and that debris left by transmutation reactions at the twin interface can accelerate formation of cracks at the twin-twin intersections.

3.3 Twin-Grain Boundary

The most important aspect of twin-GB interaction is twin-accommodation slip. Twin-accommodation slip or accommodation effects refer to slip in the parent or in the twin generated by twinning itself [1]. In fact, for single crystals (SC) materials, the shape change by a twin is partly accommodated by kinking and partly by slip [26–31]. For polycrystalline materials, it is interesting to learn that most of the shape change brought by twinning is actually accommodated by slip, because of the compatibility restrictions at the GB segments where the twin lamellae merge [32], [33]. As such, transmutation must occur early in strain starting from the first instances of twin growth. The disregard of accommodation effects in crystal plasticity models has led to a substantial underestimation of dislocation densities in the parent. For Regime II to be properly captured, it is important to capture twin-accommodation dislocations in the parent, which can be basal as well as non-basal [26]. Although the role of twin-accommodation slip in plasticity has been invoked and emphasized during the nineteen fifties and nineteen sixties, current crystal plasticity (CP) and continuum mechanics (CM) models are still oblivious to it. This disregard of accommodation effects has led to a substantial underestimation of dislocation densities in the parent, and above all, to an underestimation of the effect of transmutation at early stage of stains [26]. Slip-accommodation effects at GB bear critical consequences on damage in HCP structures [34], [35]. If slip cannot be easily activated at the GB, cracks may readily open as suggested by Rémy [15], [16]. The difficulty of slip at GBs is a genuine consideration in HCP structures since rhombohedral slip is much harder than basal and prismatic slips. In fact, Holden [26] observed non-basal twin-accommodation slip even in single crystal surfaces where the material can readily relax any strain incompatibility. Recently, Zhang et al. [35] showed via MD simulation opening of cracks in molybdenum at the triple points joining a GB upon which a residual twin impinges. Here, the effect of low GB misorientation (LGBM) is of considerable importance since it does not only affects the critical resolved shear stress (CRSS) for twinning [36–39] but also for slip [9]. In fact, these authors showed that LGBM effects explain the difference in the variation of CRSS of both slip and twinning upon changing the initial texture for a given material. A sharp rod-texture with axisymmetric [¯] fiber would have the CRSS of slip (resp. of twinning) twice softer (resp. harder) than that of a ―spotty" texture with an axisymmetric [ ] (basal) fiber. In addition to LGBM, Hondros and McLean [40] pointed out that segregation of impurities and precipitation and GBs can dramatically impede accommodation slip at twin impingements and thus cause crack to incubate.

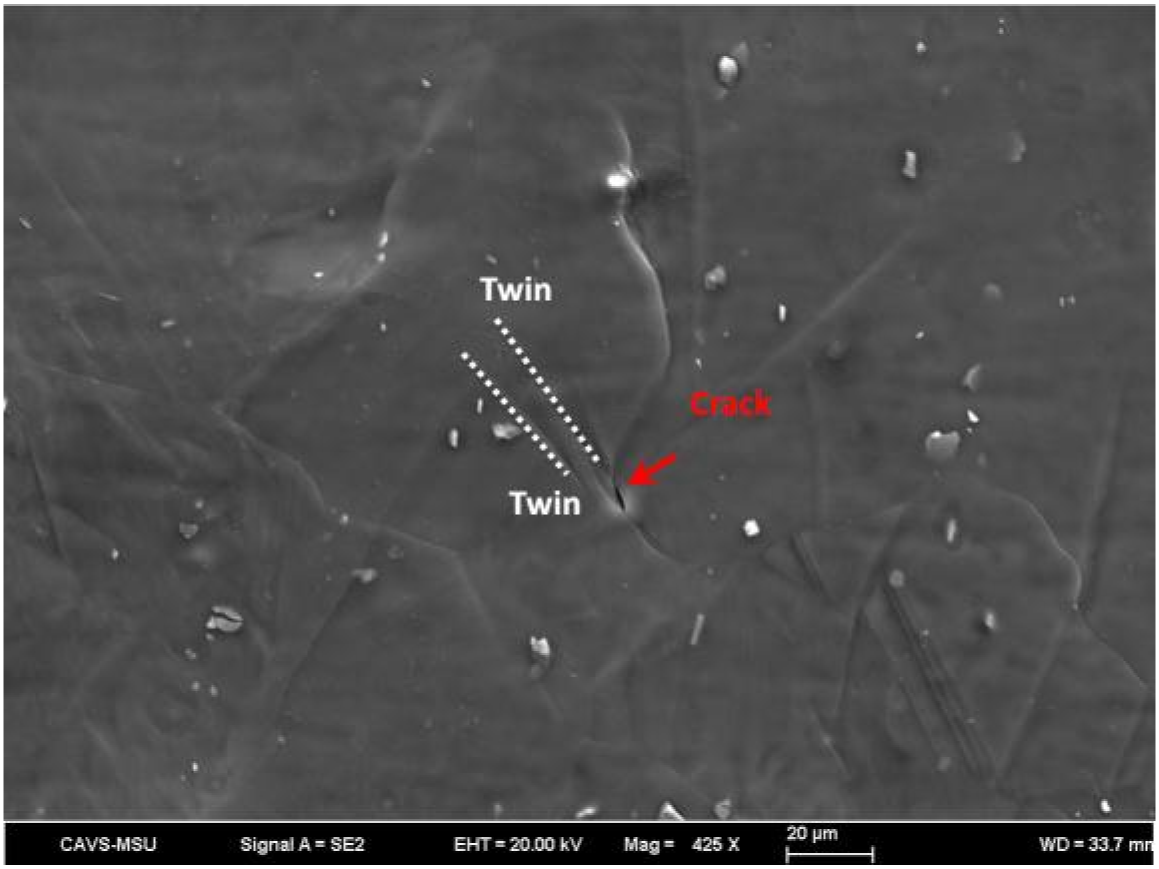

Figure 3.3 Twin-Grain Boundary Interaction Resulting in Crack Nucleation

3.4 Backstress

Due to the necessity of multiple slip and twinning modes with strong disparities in the CRSS, backstress is considerably more pronounced in HCP structures than in cubics. Backstress induced twinning explains why weak textures, and sharp textures deform under loading directions with a low Schmid's factor (SF) range for { ¯ } twinning, do not lead to a noticeable enhancement in ductility. A gap in understanding this effect has lead nowadays to expensively ineffectual alloy and process design efforts that aimed at weakening texture to prevent twinning.

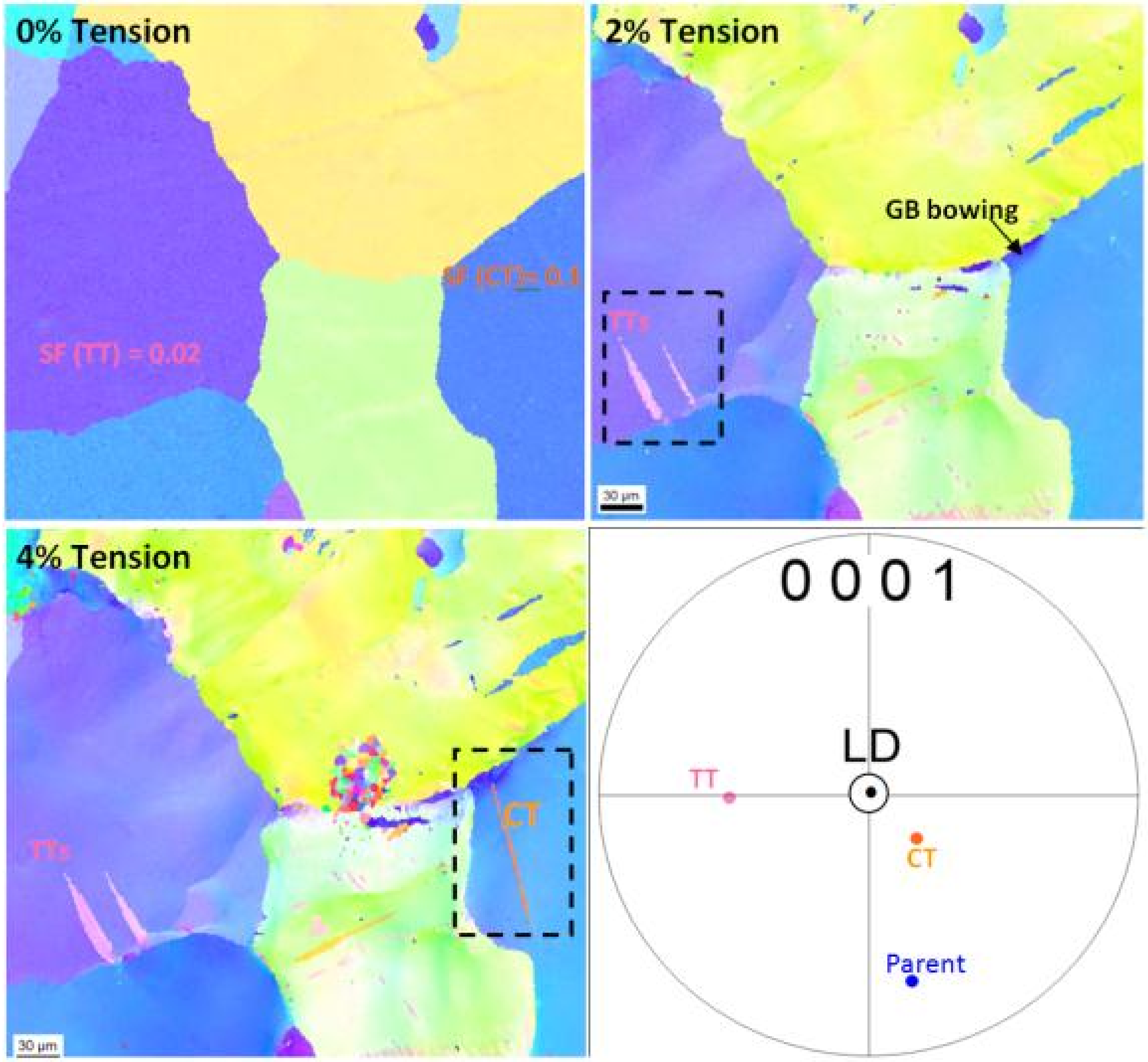

Figure 3.4 IPF maps obtained *In situ* EBSD of an AM30 alloy deformed in tension under nominal twinning conditions showing (a) initial state, (b) after 2% plastic strain with backstress-induced two { ¯ } twins nucleating at the GB separating two grains of very low SF as illustrated in –d, (c) after 4% plastic strain with backstress-induced { ¯ } twinning at the triple point, and (d) pole figure illustrating the orientation of the c-axis of the parent (blue), { ¯ } twins (magenta), and { ¯ } (orange) twins with respect to the loading direction (LD).

In situ EBSD performed on an AM30 Mg alloy loaded in tension along the main axis of the [¯] fiber show nucleation of { ¯ } twins at a GB separating two grains with almost null SF (Figures 3.4a, 3.4b and 3.4c). After 4% of plastic deformation, Figure

3.4c show nucleation of compression twinning at the triple points after substantial rotation of the grains due to non-basal slip.

3.5 Double Twinning/Contraction Twin

Although the notion of double twinning was initiated by Crocker [41], we use this terminology here to refer to any sequence of multifold twinning within an original twin [9]. In Mg alloys, double twinning is usually initiated within { ¯ } twins by { ¯ } compression twins, which readily twin by { ¯ } twinning [42]. This multifold double twinning phenomenon has been reported by many authors to play a critical role in enhancing damage in Mg[42–49]. Figures 3.5a, 3.5b, 3.6a, and 3.6b illustrate a strong correlation between crack nucleation and double and { ¯ } twins.

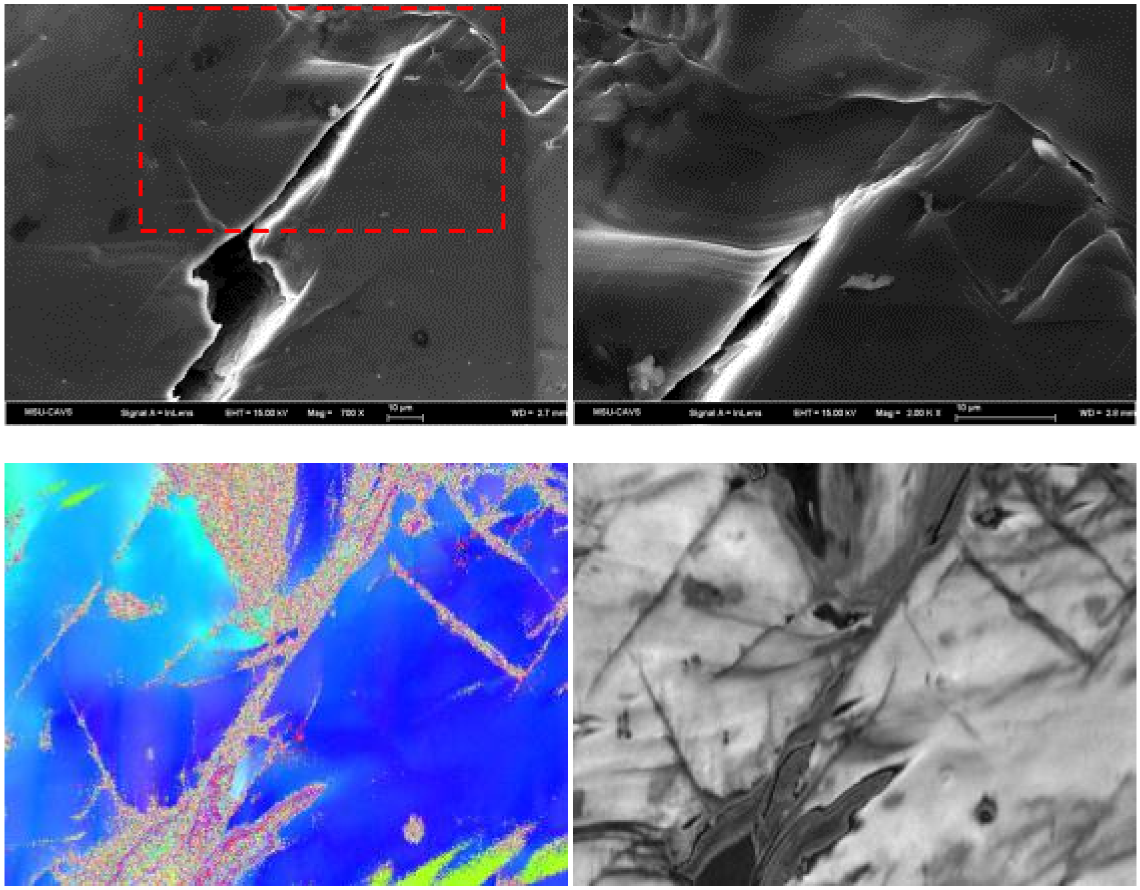

Figure 3.5 (a-b) SEM micrographs showing crack nucleation along two double twin variants exactly following follows the zig-zag meandering of the { ¯ } twins. Observe that the opening of the crack is preferentially located at the intersection of the two { ¯ } twin variants. (c) an IPF from an EBSD scan of the region highlighted in –a that couldn‘t resolve the narrow twins due to the high kinking generated at the free surface, and (d) is image quality map exhibiting better the { ¯ } twins.

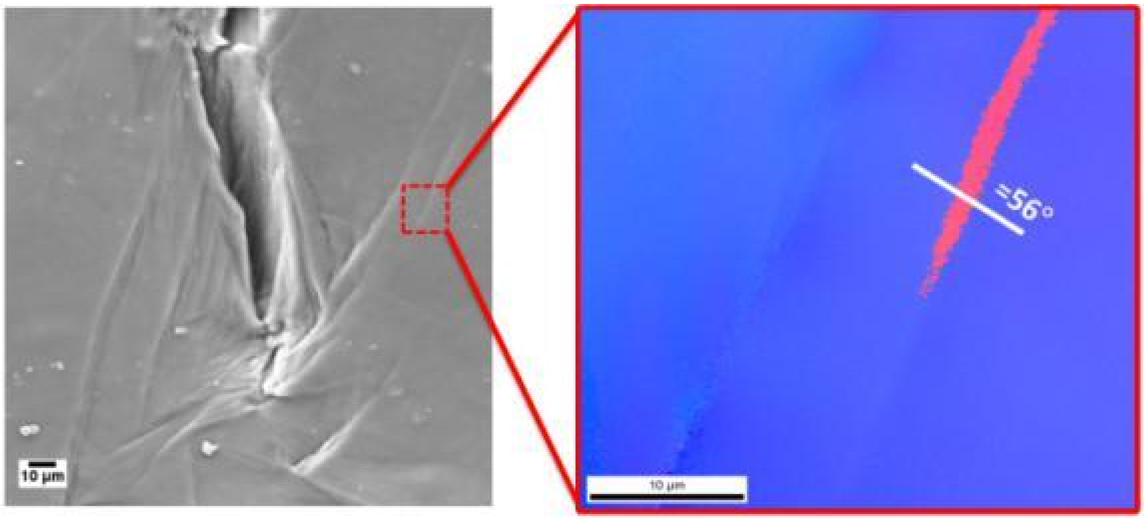

Figure 3.6 (a) an SEM micrographs showing a transgranular crack nucleating at the intersection between a tripe point and needles of { ‾ } twins, and (b) an IPF from an EBSD scan of the region highlighted in –a that resolved the needle as a { ‾ } twin inducing approximately 56° of the blue parent.

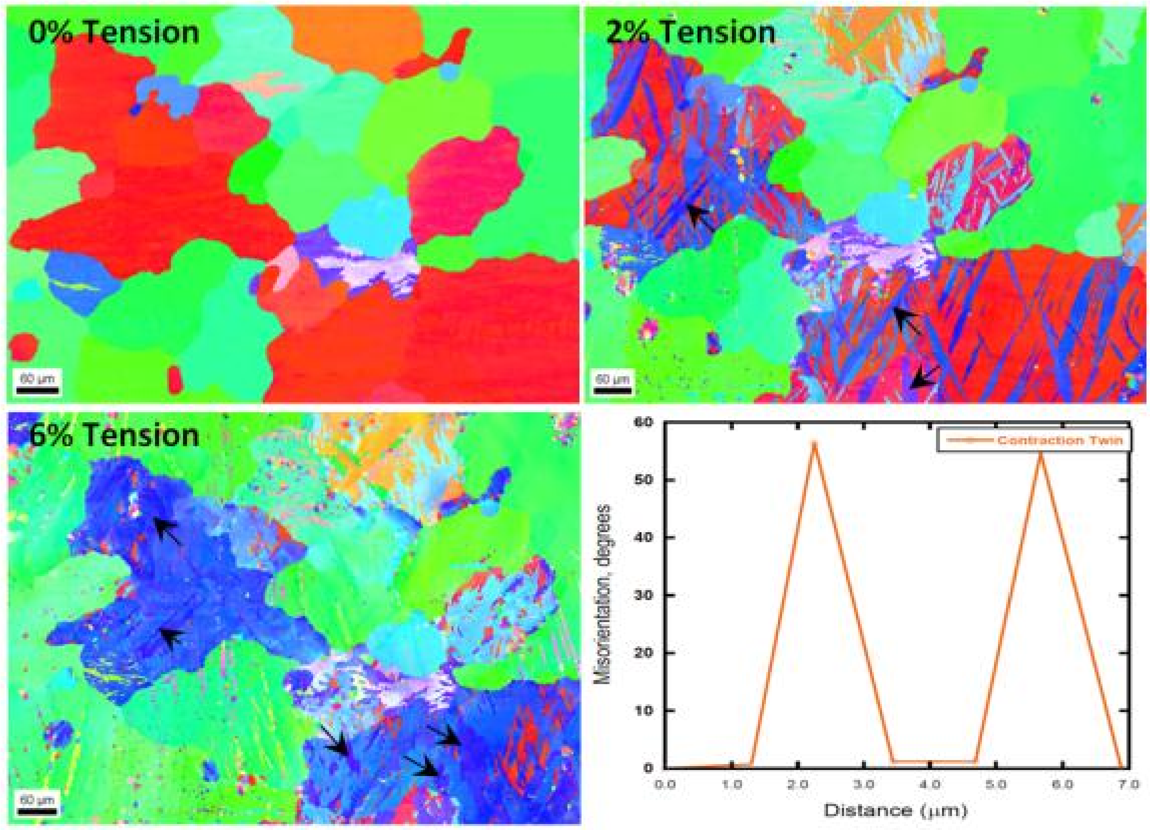

Figure 3.7 *In situ* EBSD orientation maps of an AM30 deformed in tension under profuse { ‾ } twinning conditions showing texture evolution from (a) the initial state, through (b) 2% plastic deformation, and up to (c) 6% plastic deformation with (d) a misorientation profile map across the twins marked by arrows. Massive { ‾ } i.e. double twinning (arrows) preferentially accompanying { ‾ } twins indicating a strong non-Schmid's effects. The double twins have the dark blue orientation.

Here double twinning occurred in loading directions where the SF for { ‾ } twinning is the lowest, which pinpoints again the pronounced effect of backstress on twin nucleation. Ma et al. [48] demonstrated that double twin nucleation is sensitive to strong non-Schmid's effects which pertain to the nature of prior dislocations. *In situ* EBSD maps of Figures 3.5 and 3.6 illustrate the systemic and profuse preferential nucleation of double and { ‾ } twins within twinned regions and not within a parent that has a similar SF for { ‾ } twinning.

CHAPTER IV

CONCLUSIONS

This paper successfully investigated the effect of twinning on fracture in two magnesium alloys using non-destructive electron backscatter diffraction (EBSD) on the same region. The outcome of this Master thesis was the identification of the following mechanisms that drive damage in Mg, and as such, formally explain the low ductility of this material, which hindered its integration in automotive body panels:

1. Slip-twin interactions, with a major effect of dislocation transmutation. The resultant generation of sessile dislocations and faults played a major role in rushing fracture. EBSD analyses suggested that complex twin-slip interaction promoted rapid strain hardening and crack nucleation.
2. Twin-twin interactions, These mechanisms were the most potent source of damage initiation, which inclused interaction between compression twinning and double twinning,
3. Twin-GB (Grain Boundary) interactions, which lead to backstresses that in a lot of cases could not be accommodated by dislocation slip or twinning, so cracks formed instead. The governing physics must be identified in a future work.

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