Fracture toughness of hot rolled pure magnesium: Correlation with microstructure and texture

Prakash C. Gautam¹*, H.N. Bar², S. Sivaprasad², S. Tarafder², Somjeet Biswas¹*

¹Light Metal and alloy research Lab, Department of Metallurgical & Materials engineering, Indian Institute of Technology - Kharagpur, Kharagpur, India.
²National Metallurgical Laboratory - Jamshedpur, Jamshedpur, India.

Email: pcgautam@iitkgp.ac.in, somjeetbiswas@metal.iitkgp.ac.in

Abstract. Pure magnesium possesses high specific strength, and hence it has excellent potential for biodegradable structural bioimplants. Such a load-bearing application requires the material to have sufficient fracture toughness to sustain the presence of undesirable microcracks. In this work, biomedical grade pure magnesium was hot-rolled to obtain equiaxed microstructure with basal texture. Single edge notch bending specimens with a notch along the rolling direction, transverse direction, and 45° to both were prepared. Fatigue pre-cracking was performed, followed by the fracture toughness tests. All the samples show sufficient plasticity during the fracture toughness test to evaluate J_{1C}. The characterization was done by electron backscatter diffraction. The influence of initial texture and the strain state on the evolution of twins in the vicinity of the crack path and fracture toughness was comprehended.

Keywords: Magnesium; Bioimplant; Microstructure; Texture; Fracture toughness; Twins;

1. Introduction
The demand for temporary load-bearing bio-implant has increased in recent years. From several decades, the traditional metallic bio-implants such as titanium alloys, stainless steels, cobalt-chromium, and others have been used for load-bearing applications. These are usually biocompatible but not biodegradable [1,2]. Apart from that their mechanical properties are mismatched with the natural bones. Thus, the stress shielding effects due to their high modulus, second surgical procedure and risk of toxic metal ion release may lead to several health care issues [3]. In recent year pure magnesium (Mg) and Mg alloys are being viewed as a structural load-bearing component for the bio-implant applications owing to its biocompatibility and biodegradability [4]. Moreover, Mg is a vital element for the human body, and the generated biodegradation product can be easily absorbed and metabolized by the human body. Thus, the major advantages are that there is no requirement for another surgery to remove it after the healing process [5]. Moreover, its mechanical properties are well-matched with the natural bones. It possesses elastic moduli of about 40-45 GPa which is much closer to that of the natural bone. Thus, it has the potential for avoiding the stress shield [2-5]. Apart from that, it has several striking properties such as low density and higher specific strength [3-6]. Despite owing such remarkable properties, its application is still limited for bio-implant due to its poor strength and fracture toughness [6]. Thus, the primary focus on improving the fracture toughness is needed. Additionally, Mg is highly anisotropy in nature due to the activation of only two independent basal slips at ambient temperature, as its critical resolved shear stress (CRSS) is much lower than that of non-basal slips. Hence the governing basal slips produce sharp basal texture during processing that produces high plastic anisotropy at ambient temperature. Consequently, its application potentials are further impeded [7-9]. Thus, the analysis of
fracture toughness in various directions need to be elaborated before using it as bio-implant, considering the possibilities of various loading configuration inside the material.

2. Experimental procedure

Biomedical grade pure Mg (99.94% purity) was received in the form of the hot-rolled plate to determine its fracture toughness. Prior to fracture toughness testing, the microstructure and texture observation were carried out on the rolling plane by using electron backscattered diffraction technique. Further, yield strength (YS) and tensile strength (TS) was obtained by ambient temperature tensile testing of the miniature sample obtained from the hot-rolled plate. Single edge notch bend (SENB) specimen were prepared as per ASTM E-1820 [10] for the fracture toughness test. Three different samples were prepared from the so obtained rolled plate; notch length parallels to RD, perpendicular to RD, and 45° to RD to measure the variation in fracture toughness in different directions. A fatigue pre-crack length of 0.5 W was introduced prior to the fracture toughness test. This was done at a frequency of 5 Hz and a constant stress intensity factor range (ΔK) of 3 MPa√m so that the maximum load corresponding to this ΔK value could be kept below $P_f = \frac{0.5 B b}{s} \sigma_Y$ as per ASTM E-1820 [10]. Where B is the thickness, b is the uncracked ligament, $(w - a_0)$, $\sigma_Y = (\sigma_{YS} + \sigma_{TS})/2$, and S is the span length. After the introduction of fatigue pre-crack, the specimens were loaded to determine the fracture toughness. The microstructure of the fracture sample was examined in the plane containing crack propagation by using an optical microscope. The fracture surface was observed using SEM.

3. Result and discussion

3.1. Initial material

The inverse pole Figure (IPF) map of the as-received material and their grain size distribution is shown in Fig. 1. The as-received hot-rolled Mg mostly contains large equiaxed grains with an average AF (NF) grain size of ~51.4 (~12) μm. The microstructure portrays the presence of mostly basal orientation in the rolling plane. The texture of the as-received material is plotted in terms of (0002), (101̅0) and (112̅0) pole figures and are shown in Fig. 2. The texture shows the (0002) poles parallel to the plane perpendicular to the ND with {101̅0} and {112̅0} fibre axis parallels to the rolling direction (RD). This indicates the formation of sharp basal fiber texture as a result of a hot rolling operation [6,11].

![Fig. 1. IPF map of hot rolled Mg and their grain size distribution.](image)

The deformation texture after the hot rolling of Mg is (0002) <112̅0> as reported in the literature [6]. However, during the dynamic recrystallization, the crystal rotates from <112̅0> to <101̅0> i.e., 30° around the c-axis [8,11-14]. Therefore, the recrystallization texture in the Mg is represented by (0002) <101̅0>. Hence, from the pole figure analysis, deformation, as well as recrystallization texture, could be observed representing that the as-received sample was dynamically recrystallized.
Fig. 2. (0002) (10\̅10) and (11\̅20) pole Figures of the hot-rolled Mg with their reference direction and the intensities.

### 3.2. Fracture toughness

The load-displacement curve during the fracture toughness test for all the samples showed non-linear behavior before it reaches the maximum load. The maximum load (P_max) was obtained during the test for all the samples. Further, a load value (P_Q) was obtained from an intersection of a 95% secant line (from the origin) with the load-displacement curve. The ratio between P_max and P_Q was evaluated, which was found greater than 1.11. Thus, it suggested that the samples had undergone enough plastic deformation during the loading. Moreover, the dimensions required for the test to have a plane strain fracture toughness was below its minimum value according to the following equation.

\[ a, B, W - a \geq 2.5\left(\frac{K_Q}{\sigma_{ys}}\right)^2 \]  

Therefore, the test was not valid to determine the \( K_{1C} \) [10]. However, the elastic-plastic fracture toughness (\( J_C \)) values were obtained for all the samples. In this regard, the provisional fracture toughness (\( K_Q \)) value was obtained by using the formula given below for the SENB specimen as per ASTM-1820 [10].

\[ K_Q = \left[\frac{P_Q S}{BW^{1.5}}\right] f \left(\frac{a}{W}\right) \]  

where \[ f \left(\frac{a}{W}\right) = \frac{2 \left(\frac{a}{W}\right)^{0.5} \left[1.99 - \left(\frac{a}{W}\right)\left(1 - \frac{a}{W}\right)\left(2.15 - 3.93\left(\frac{a}{W}\right) + 2.7\left(\frac{a}{W}\right)^2\right)\right]}{2(1 + 2\frac{a}{W})(1 - \frac{a}{W})^{1.5}} \]  

Now the \( J_C \) was calculated as follow;

\[ J_C = J_{el} + J_{pl} \]  

i.e. \( J_C = K_Q^2 \left(1 - v^2\right)/E + J_{pl} \)  

\[ J_{pl} = \frac{2A_{pl}}{B_N b_0} \]  

\( A_{pl} \) is the area enclosed by the load-displacement curve and the line drawn parallel to the initial linear part of load-displacement curve. All the calculations were done to get \( K_Q \) and \( J_C \) values for all the samples. The values are shown in Table 1 below. Now the minimum thickness required to have a size-independent elastic-plastic fracture toughness (\( J_{1C} \)) was achieved for all the samples as per the expression below [10].

\[ B, W - a = 25 \frac{J_C}{\sigma_{ys}} \]  

From the calculated data, the provisional fracture toughness (\( K_Q \)) value is found maximum for the \( a_n - 45^\circ \) to RD configuration, followed by \( a_n \perp RD \) and \( a_n \parallel RD \). However, the elastic-plastic fracture toughness (\( J_{1C} \)) is in the order; \( a_n \parallel RD \) > \( a_n \perp RD \) > \( a_n - 45^\circ \) to RD. From the pole figure analysis, it could be noticed that along the RD; the maximum intensities are contributed by <10\̅10> directions. Whereas, along TD, and 45° to RD, maximum intensities are contributed by <11\̅20> directions.
| Notch length to RD | $P_{\text{max}}$ (N) | $P_Q$ (N) | $P_{\text{max}}/P_Q$ | $K_Q$ (MPa$\sqrt{m}$) | $A_{pl}$ (Nmm$^2$) | $J_{pl}$ (N/m) | $J_{IC}$ (N/m) |
|-------------------|-------------------|-----------|-----------------|-----------------|----------------|-------------|-------------|
| Parallel          | 374               | 255       | 1.46            | 5.42            | 58             | 3.86        | 4.15        |
| Perpendicular     | 368               | 279       | 1.31            | 5.98            | 49.4           | 3.29        | 3.61        |
| 45 degree         | 370               | 325       | 1.13            | 6.92            | 32             | 2.13        | 2.61        |

Thus, it could be concluded here that the energy required to propagate the crack along the $<10\overline{1}0>$ is more than along the $<11\overline{2}0>$ direction. The close-packed directions are actively distributed perpendicular to and 45° to the RD as a result of dynamic recrystallization [15]. In general, dislocations easily glide on the close-packed plane and directions as deformation proceed. Therefore, it is reasonable to postulate that the pre-crack having notch length 45° to the RD and perpendicular to the RD are easy to propagate [16].

3.3. Fractography

The typical SEM images of the fracture samples for all the samples (a) Notch parallel to RD, (b) Notch perpendicular to RD, and (c) Notch 45° to RD was obtained after the fracture toughness test. Fig. 3 shows the fracture surface in the rapid fracture, i.e., beyond the fatigue pre-crack region. All the samples show a combination of ductile and brittle fracture behavior. Overview of the fracture surface reveals that significant fracture area is dominated by shear lip along with cleavage fracture surface associated with river pattern.

![SEM images](image_url)

Fig. 3. SEM image of the fracture surface for the samples with their notch parallel, perpendicular, and 45° to the rolling direction.

The fracture surface of $a_n \parallel$ RD contains shallow dimples, representing enough plasticity during the fracture. The level of plastic deformation at the crack-tip produces the hindrance to the fracture because typical dimples decide the amount of plastic deformation at the crack-tip [16-18]. The fracture surface of $a_n \perp$ RD is dominated by cleavage fracture with some shear lip. However, the fracture surface of $a_n \perp$ 45° to RD comprises of shallow dimples with few shear lips and cleavage fracture.

3.4. Optical microstructure

Fig. 4 shows the optical microstructure of the fracture samples in the plane perpendicular to ND. All the samples show the formation of twins of thin lamellar structure in the vicinity of the crack path. This
could be possible because of the establishment of the stress triaxiality in the plastic zone at the crack tip. In this regard, various types of twins could form in the region of plastic zone size, depending upon the triaxial state of the strain component corresponding to the various stress configuration on the crack tip [18,19]. The amount of twin formation can be seen maximum for the sample \( a_n \parallel RD \) followed by \( a_n \perp RD \) and \( a_n 45^\circ \) to RD. Thus, in this context, the amount of twin formation can be well correlated with the fracture toughness of the samples. In this work, the fracture toughness is found to be increased with the increase in twin formation.

The present result is supported by previous works [17,18], where they have shown that the energy required for the separation of two surfaces increases with the formation of twins. From Fig. 4, various twin structure such as parallel and crossing type of morphology is observed. Twinning leads to a change in the crystallographic orientation, which may lead to the textural hardening [20,21]. Besides, the dislocations produced within the parent grains can get pinned at the twin and the twin-twin intersection boundaries. Further, within the twin domain, slips can get activated. Therefore, the dislocation-dislocation interaction and pile-up of dislocations at the twin boundaries lead to the strain hardening. Thus, it increases the work of separation [17,18,20,21].

![Fig. 4. The optical image in the vicinity of a crack path for the samples with their notch parallel, perpendicular, and 45° to the rolling direction.](image)

### 4. Conclusions

1. The elastic-plastic fracture toughness \( (J_{IC}) \) was found maximum in the orientation having notch length parallel to rolling direction followed by notch length perpendicular to the RD and notch length 45° to the RD.
2. From the texture analysis, it could be perceived that the energy required to propagate the crack along the \(<10\overline{1}0>\) directions are more; as a result, the higher fracture toughness is found along the notch length parallel to RD.
3. The fracture surface comprises of mixed fracture behavior, revealing that the significant fracture area is dominated by shear lip along with cleavage fracture surface associated with river pattern.
4. Fracture toughness was found to be increased with the increase in the number of twins.

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References

[1] Ren L and Yang K (2013) Bio-functional design for metal implants, a new concept for development of metallic biomaterials J Mater. Sci. Technol. 29 1005–10
[2] Cifuentes S C, Gavilán R, Lieblich, Benavente R and González-Carrasco J L (2016) In vitro degradation of biodegradable polylactic acid/magnesium composites: Relevance of Mg particle shape Acta Biomater. 32 348–57
[3] Cai C, Song R, Wen E, Wang Y and Li J (2019) Effect of microstructure evolution on tensile fracture behavior of Mg-2Zn-1Nd-0.6Zr alloy for biomedical applications Mater. Des. 108038
[4] Zhang E, Xu L and Yang Y (2005) Formation by ion plating of Ti-coating on pure Mg for biomedical applications. Scripta Mater 53 523–27
[5] Li Z, Gu X, Lou S and Zheng Y (2008) The development of binary Mg-Ca alloys for use as biodegradable materials within bone Biomater. 29 1329–44
[6] Estrin Y, Nene S S, Kashyap B P, Prabhu N and Al-Samman T (2016) New hot rolled Mg-4Li-1Ca alloy: A potential candidate for automotive and biodegradable implant applications Mater. Lett. 173 252–56
[7] Agnew S R and Duygulu Ö (2005) Plastic anisotropy and the role of non-basal slip in magnesium alloy AZ31B Int. J. Plast. 21 1161–93
[8] Biswas S, Singh Dhinwal S and Suwas S (2010) Room-temperature equal channel angular extrusion of pure magnesium Acta Mater. 58 3247–61
[9] Miller V M, Berman T D, Beyerlein I J, Jones J W and Pollock T M (2016) Prediction of the plastic anisotropy of magnesium alloys with synthetic textures and implications for the effect of texture on formability Mater. Sci. Eng. A 675 345–60
[10] ASTM E1820 Standard Test Method for Measurement of Fracture Toughness E1820 2017 1–53
[11] Biswas S, Singh D S, Beausir B, Toth L S and Suwas S (2015) Thermal Response on the Microstructure and Texture of ECAP and Cold-Rolled Pure Magnesium Metall. Mater. Trans. A 46 2598–13
[12] Biswas S, Dhinwal S S, Bhowmik A and Suwas S (2008) Study of Texture Evolution of Pure Magnesium during ECAE Using EBSD Mater. Sci. Forum 584–586 343–48
[13] Biswas S, Gautam P C, Shukla A J and Chouhan D K (2020) Dynamic recrystallization and its effect on microstructure and texture evolution in magnesium alloys Refer. Module Mater. Sci. Mater. Eng. B 978-0-12-815732-9.00016-4
[14] Biswas S, Beausir B, Toth L S and Suwas S (2013) Evolution of texture and microstructure during hot torsion of a magnesium alloy Acta Mater. 61 5263–77
[15] Wu W X, Jin L, Wang F H, Sun J, Zhang Z Y, Ding W J and Dong J (2013) Microstructure and texture evolution during hot rolling and subsequent annealing of Mg–1Gd alloy Mater. Sci. Eng. A 582 194–02
[16] Somekawa H and Mukai T (2006) Fracture toughness in a rolled AZ31 magnesium alloy. J. Alloys Comp. 417 209–13
[17] Verma R, Srinivasan A, Nath S K and Jayaganthan R (2018) Tensile and fracture toughness behaviour of ultrafine grained Mg-4Zn-4Gd alloy processed through hot rolling followed by hot pressing Mater. Sci. Eng. A 742 318–33
[18] Prasad N S, Naveen Kumar N, Narasimhan R and Suwas S (2015) Fracture behavior of magnesium alloys - Role of tensile twinning Acta Mater. 94 281–93
[19] Kondori B and Benzerga A A (2014) Effect of stress triaxiality on the flow and fracture of Mg alloy AZ31 Metall. Mater. Trans. A 45 3292–07
[20] Sahoo S K, Toth L S and Biswas S (2019) An analytical model to predict strain-hardening behaviour and twin volume fraction in a profoundly twinning magnesium alloy Int. J. Plast. 119 273–90
[21] Sahoo S K, Biswas S, Toth L S, Gautam P C and Beausir B (2020) Strain hardening, twinning and texture evolution in magnesium alloy using the all twin variant polycrystal modelling approach. Int. J. Plast 102660