

# 1       **Microstructure, texture and tensile properties of ultrafine/nano grained** 2       **magnesium alloy processed by accumulative back extrusion**

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## 9       **Abstract**

10               An AZ31 wrought magnesium alloy was processed by employing multipass accumulative  
11 back extrusion process. The obtained microstructure, texture and room temperature tensile  
12 properties were characterized and discussed. Ultrafine grained microstructure including nano  
13 grains were developed, where the obtained mean grain size was decreased from 8 to 0.5  $\mu\text{m}$  by  
14 applying consecutive passes. The frequency of both low angle and high angle boundaries  
15 increased after processing. Strength of the experimental alloy was decreased after processing,  
16 which was attributed to the obtained texture involving the major component lying inclined to the  
17 deformation axis. Both the uniform and post uniform elongations of the processed materials were  
18 increased after processing, where a total elongation of 68 pct was obtained after six-pass  
19 deformation. The contribution of different twinning and slip mechanism was described by  
20 calculating corresponding Schmid factors. The operation of prismatic slip was considered as the  
21 major deformation contributor. The significant increase in post uniform deformation of the  
22 processed material was discussed relying on the occurrence of grain boundary sliding associated  
23 with the operation of prismatic slip.

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25 **Introduction**

26           Magnesium and its alloys exhibit poor formability and negligible hardenability at room  
27 temperature, which impede their potential application for automobile, electronic and aerospace  
28 industries [1]. Thus, to employ their merits over many structural materials including superior  
29 specific stiffness and strength, the low temperature hardenability and ductility of magnesium  
30 alloys should be improved. Two promising ways in this direction are the grain refinement [2, 3]  
31 and manipulation of crystallographic texture [4, 5]. In large-grain magnesium, basal slip  
32 facilitates microscopic yielding at very low stress. In order to accommodate further deformation,  
33 other slip systems should be activated. Koike et. al. [6] showed that, in fine grained magnesium,  
34 the dislocation cross-slip to non-basal planes occurred at very low stress level due to grain  
35 boundary compatibility stress. The substantial dislocation activity on non-basal planes brought  
36 about a respectable room temperature tensile ductility. Moreover, the low symmetry HCP crystal  
37 structure makes magnesium alloys very sensitive to crystallographic texture in such a way that  
38 their mechanical properties vary considerably with changes in sample orientation [7, 8].  
39 Activation of different slip and twinning systems is primarily influenced by the initial  
40 orientation. For ductilizing magnesium the initial texture should be tailored ensuring that the  
41 Schmid factor (SF) for the basal slip is maximized [9].

42           Severe plastic deformation (SPD) processes are considered to improve room temperature  
43 mechanical properties in magnesium alloys, e.g. [5, 10]. During SPD, the initial grain size of the  
44 material is significantly reduced by thermally aided plastic deformation through different  
45 recrystallization mechanisms [10, 11].

46 As deformation and grain refinement may also cause variations in crystallographic  
47 texture, the post-SPD mechanical properties result from combined effects of grain refinement  
48 and texture changes. Furthermore, it has been demonstrated that the degree of grain refinement  
49 by SPD, itself, is strongly coupled to the development of texture and substructural evolutions  
50 [12, 13]. It has been proposed that grain refinement is primarily the result of the interaction of  
51 shear plane with texture and the crystal structure, followed by an influence coming from the  
52 accumulation of shear strain during severe deformation [12]. It has been known that moderate to  
53 strong level of complex textures can be induced during SPD of magnesium alloys [14, 15].

54 SPD methods have been examined in several studies to explore the possibility of  
55 inducing improved low temperature strength and ductility in magnesium alloys, e.g. [16].  
56 Reported results on the mechanical properties of SPDed magnesium alloys illustrated that a  
57 miscellaneous combination of strength and ductility alterations may be obtained, depending on  
58 synergistic or antagonistic effects of grain refining and texture change [17-20]. Furthermore it  
59 was recognized that the Hall–Petch parameters are texture dependent so that easy activation of  
60 basal slip introduces lower values, while inhibition of basal slip leads to higher values [20]. Xing  
61 et. al. [21] and Zuberova et. al. [22] reported that SPD of AZ31 alloy gave rise to an increased  
62 strength accompanied by a minor ductility. The latter observations describe a case where the  
63 strengthening due to grain refinement is amplified by texture hardening, while the latter  
64 counteract the ductulizing effect of former. However, results of opposite trend were obtained by  
65 Kim et al. [23] where the yield stress of a wrought AZ61 alloy processed by eight equal channel  
66 angular pressing (ECAP) passes dropped appreciably as compared to the initial material, while  
67 the tensile ductility was significantly increased. Dissimilarly, the results reported by Miura et. al.

68 [24] on multi-axial forging of AZ61 alloy implied that although the tensile strength increased  
69 with a decrease in grain size and texture changes, the ductility was not spoiled.

70 It is generally accepted that grain refinement leads to a decrease in strain hardening as  
71 yield strength is increased [25], however, Ghosh and Yang [26] believed that the combined effect  
72 of increased strain rate sensitivity and strain hardening may offset the loss in ductility in AZ31  
73 magnesium alloy. In addition, microstructure features, such as grain boundary nature and  
74 dislocation structures, could have important effects on mechanical properties. Figueiredo et. al.  
75 [19] showed that the occurrence of grain boundary sliding (GBS) plays a key role in low strain  
76 rate deformation of fine grained pure magnesium. However, Wang and Ma [25] suggested that  
77 the design and processing of a bimodal or even multi-modal grain structure is a profitable  
78 approach for developing new materials. They made the point that a uniform and extremely fine  
79 nanocrystalline structure is neither necessary nor beneficial for properties and applications, if a  
80 high ductility, in addition to the high yield strength is desired. Though the reported works  
81 accentuated on the effect of texture changes and grain refinement on the final properties, rare  
82 research could be found in the literature dealing with the detailed discussion on the role of  
83 different deformation mechanisms as well as microstructure refinement on the mechanical  
84 properties of magnesium SPD products.

85 A new continuous SPD process, the so-called accumulative back extrusion process  
86 (ABE), which is appropriate technique for production of ultrafine/nano grained bulk material has  
87 been introduced in recent years [27-29]. Previous works mainly dealt with the developed strain  
88 patterns [30], microstructural evolutions [29], homogeneity [31] and texture evolution [32]  
89 during ABE processing. In this work the tensile properties of an AZ31 magnesium alloys  
90 processed by accumulative back extrusion (ABE) was examined. Moreover, the effect of

91 microstructure and texture evolution on the final mechanical properties of the ultra/fine grained  
92 magnesium was described relying on the contribution of different deformation mechanisms.

93

## 94 **2. Experimental procedure**

95 A commercial AZ31 alloy (Mg-2.9Al-0.9Zn-0.7Mn, %wt), received in the form of rolled  
96 plate, was used as experimental alloy. Cylindrical samples for ABE processing were machined  
97 with the dimensions of  $H8 \times \Phi 18 \text{ mm}^2$ . Briefly, the first step of ABE consists of the back  
98 extrusion of the workpiece into the gap between the inner punch and the die. In the second step  
99 the back extruded material is forged back to the initial cross section by the outer punch  
100 completing one ABE cycle. The previous work showed that an average equivalent strain of 2 is  
101 induced during each step [30]. ABE process was conducted at a ram speed of 5 mm/min and  
102 temperature of 553 K (280°C). According to the relatively low stroke speed the temperature  
103 increasing during extrusion was ignored. The  $\text{MoS}_2$  spray was used to reduce the friction  
104 between the work piece and the tool surfaces. The initial material showed a mean grain size of 25  
105  $\mu\text{m}$  (Fig. 1a). Crystallographic texture measurements were conducted using X-ray diffraction in  
106 the reflection geometry with a four circle goniometer and Cu K $\alpha$  radiation. Experimental (10–  
107 10), (0002), and (10–11), pole figures were collected on a  $5^\circ \times 5^\circ$  grid for sample tilts,  $\alpha = 0\text{--}85^\circ$ ,  
108 and azimuthal rotations,  $\varphi = 0\text{--}355^\circ$ . Background and defocusing corrections were made using  
109 experimentally determined data from random powder samples. The texture of initial material was  
110 illustrated in Fig. 1b, where a strong fiber texture with most of the basal planes aligned parallel  
111 to the rolling direction (RD)–transverse direction (TD) plane. For all the ABE samples, the  
112 center of the rectangular cross section of ABEed sample was considered for representative  
113 metallographic observations. The microstructures of the deformed material were examined

114 through optical microscopy and transmission electron microscopy (TEM). The TEM  
115 investigation was performed using a Philips CM 20 microscope working at 200 kV. Toward this  
116 purpose, a slice of the ABEed workpieces was first cut parallel to the extrusion direction. The  
117 slice was then thinned by grinding to a sheet with 100  $\mu\text{m}$  thickness. The TEM samples were  
118 then mechanically polished using a twin-jet polishing unit. The perforation was done by a  
119 solution of 1% perchloric acid, 99% ethanol at a polishing temperature of 275 K. Electron back  
120 scattered diffraction (EBSD) was performed using scanning electron microscope (FEGSEM,  
121 Zeiss Ultra Plus) equipped with EBSD camera. The microstructural maps were derived from  
122 EBSD, which shows grains and sub-grains based on misorientation angle on adjacent points. To  
123 analysis microstructure, three scans were made over the cross section.

124 Because of the small size of the ABEed workpiece, miniaturized specimen were  
125 machined from the as-received and ABE processed materials for tension testing with a gage  
126 section of  $0.7 \times 1 \times 2$  mm, the longitudinal direction of which was selected to be parallel to the  
127 ABExtrusion (as shown in Fig. 2a). For the as-received material the tension samples were cut  
128 along the rolling direction. Tensile tests were performed on the samples using a Deden tension  
129 machine (Fig. 2b) with a constant crosshead velocity of 0.02 mm/min providing an initial strain  
130 rate of about  $17 \times 10^{-5} \text{ s}^{-1}$ . To verify the reproducibility of the results, three tension specimens  
131 were cut from the ABEed samples and tested.

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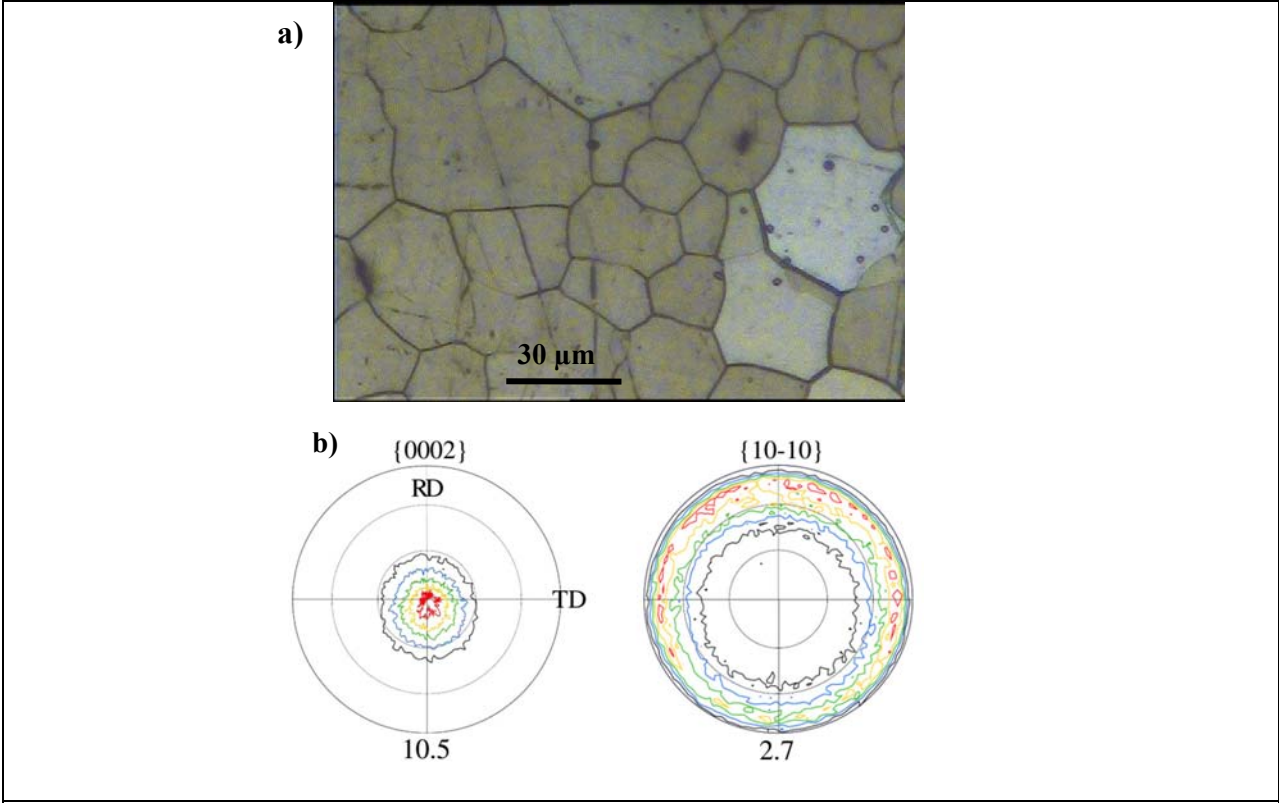
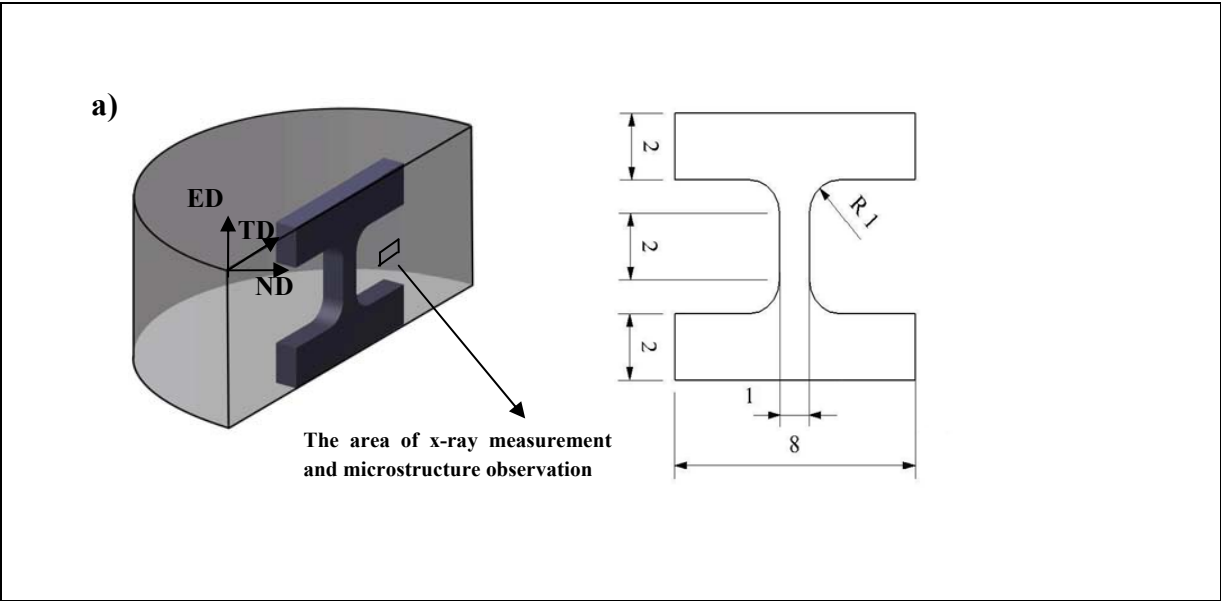


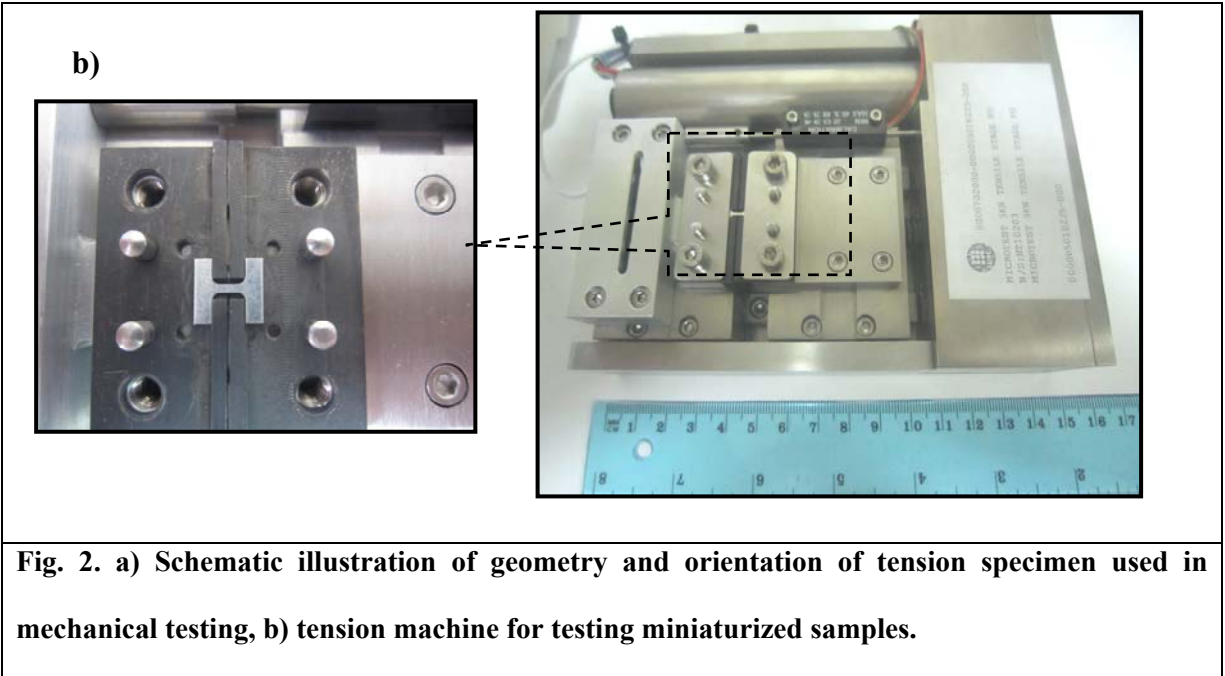
Fig. 1. a) Initial microstructure, b) measured pole figures of the experimental as-rolled AZ31 alloy.

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**Fig. 2. a) Schematic illustration of geometry and orientation of tension specimen used in mechanical testing, b) tension machine for testing miniaturized samples.**

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137 **Results and discussion**

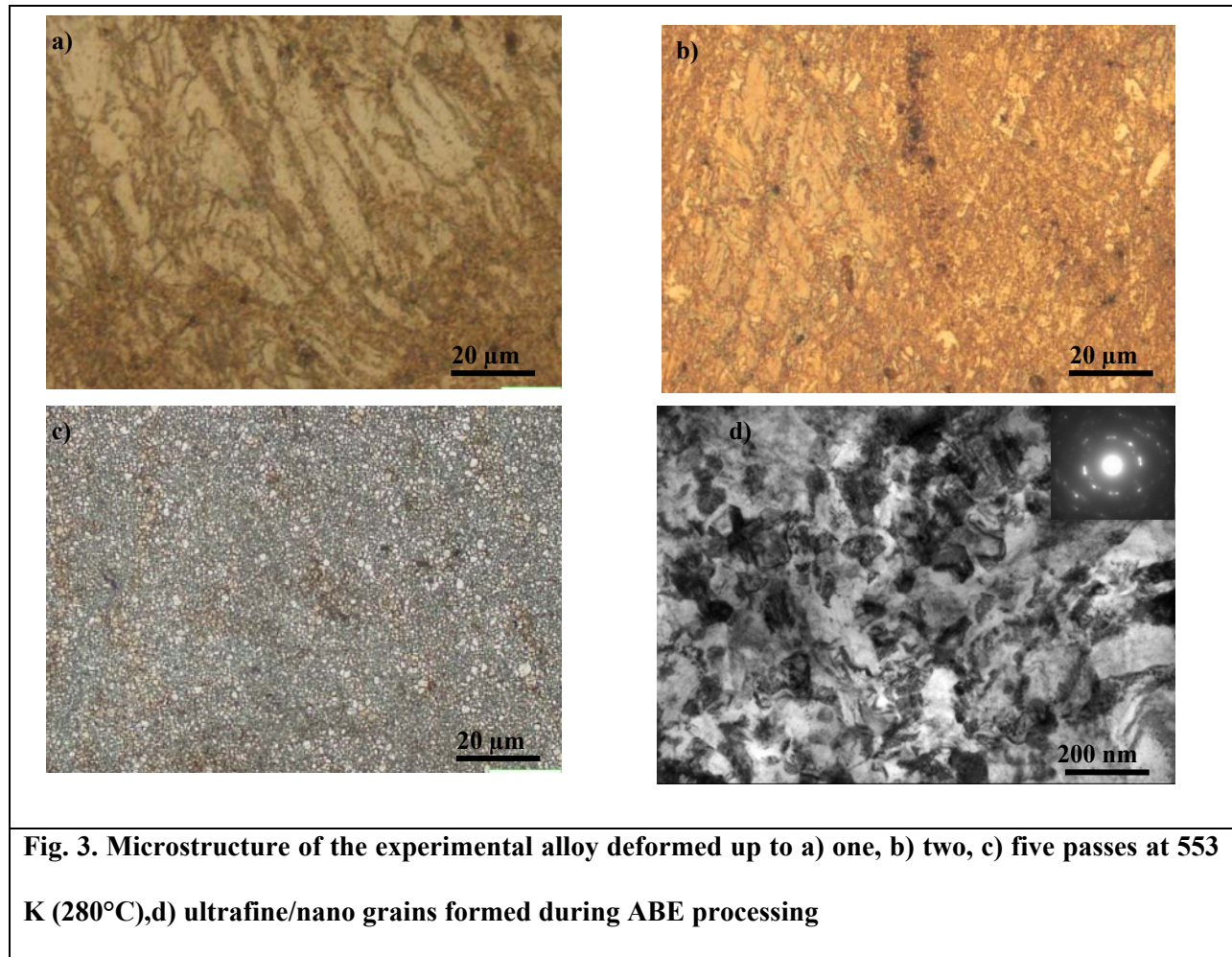
138 The microstructural observations demonstrated that an outstanding grain refinement took  
 139 place through ABE processing, where a relatively homogeneous grain refinement was achieved  
 140 after sixth pass. TEM observation showed that recrystallized areas include grains of 80 nm to 1  
 141  $\mu\text{m}$ . Figs. 3a, b, and c typically show the microstructure of the ABE processed AZ31 alloy up to  
 142 one, two and six passes, respectively. The ultrafine/nano grains developed during deformation  
 143 are depicted in Fig. 3d. As the experimental alloy was processed by successive passes the coarse  
 144 grain was substituted by fine recrystallized grains and more homogenous microstructure was  
 145 obtained. The average grain sizes were measured to be about 8, 2 and 0.5  $\mu\text{m}$  after processing of  
 146 one, two and six ABE passes, respectively. Fig. 4 displays typical grain size distribution of the  
 147 processed materials. As discussed in Ref. [11], discontinuous dynamic recrystallization was  
 148 found as the dominant grain refinement mechanism in AZ31 experimental alloy during the early



149 ABE pass, thereafter the dynamically recrystallized grains, in turn, were repetitively refined  
150 through continuous dynamic recrystallization upon applying successive passes.

151 To understand the evolution of substructure and development of fine grains during  
152 successive ABE passes, EBSD maps of the material deformed up to different ABE passes were  
153 obtained from ED-TD plane. The results are presented in Fig. 5., where formations of new grains  
154 as well as fragmentation of prior grains by subboundaries are evident. Fig. 6 shows the  
155 distributions of the misorientation angles in terms of cumulative frequency (frequency of and  
156 below a given misorientation angle) of low angle boundaries (LABs) and high angle boundaries  
157 (HABs) in the samples ABEed at 553 K (280°C). It indicates that the frequency of LABs with  
158 misorientation angles below 15° increased after single ABE, which confirms the operation of  
159 extensive dynamic recovery. However, the frequency of LABs decreases but that of HABs  
160 increases with applying successive passes. The decrease in LABs (misorientation below 15°)  
161 may indicate the transformation of LABs to HABs by incorporating the dislocations that  
162 generate during deformation. The latter requires new grain boundary area to be continuously  
163 created during deformation [33]. This is consistent with the increase in frequency of HABs.  
164 Previous results showed that the microstructure are repetitively refined through subdivision of the  
165 DRXed grains into finer ones [11].

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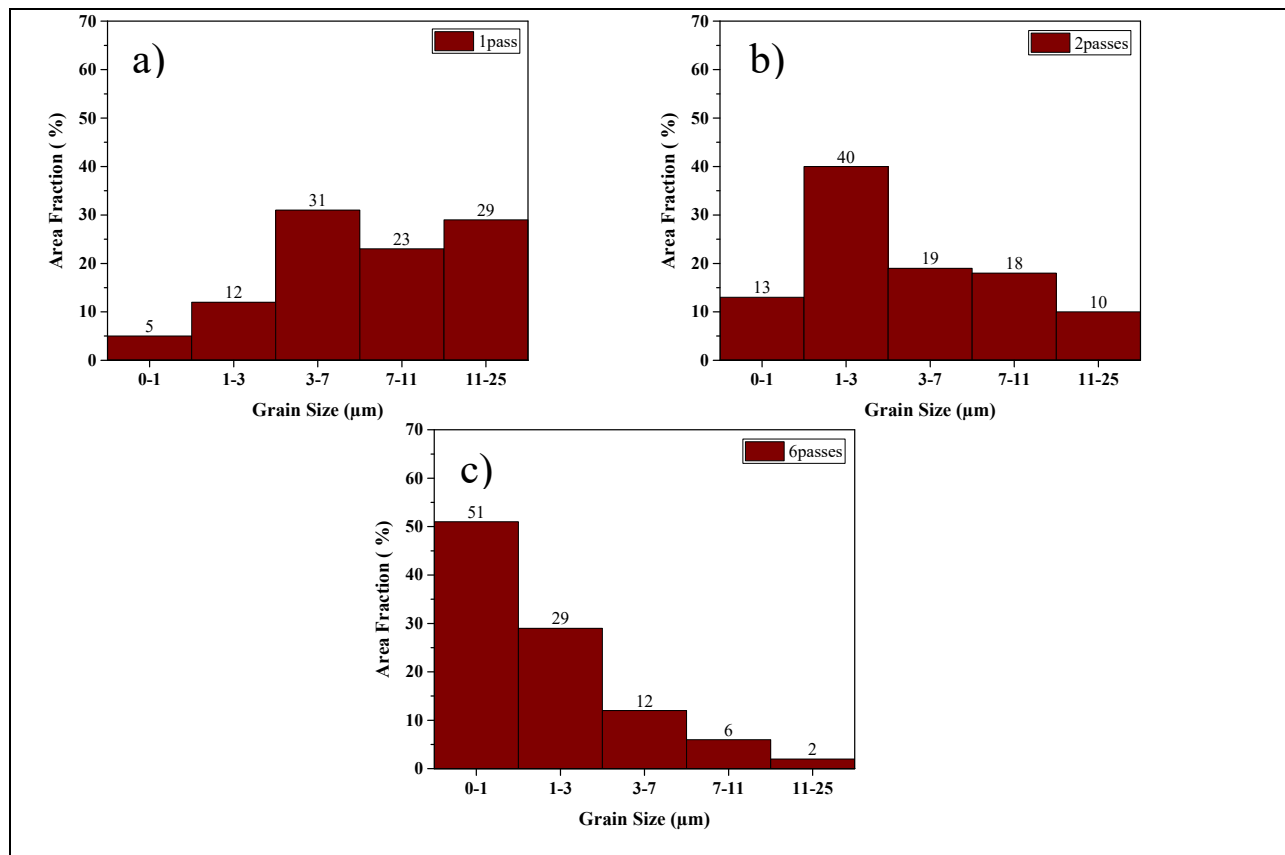
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The basal, prismatic and pyramidal pole figures of the sample deformed by different ABE passes were measured. The results were presented elsewhere [32], however, for the sake of convenience, the pole figures from the materials after the first and fourth passes are including in Fig. 7. As realized the original texture (Fig. 1b) was completely replaced and a new texture developed during the first pass. This is connected to the high shear strain magnitude imposed during ABE deformation [30]. The obtained ABE texture corresponds to the basal poles lying  $\sim 40^\circ$  away from the TD in the ED–TD plane and  $66^\circ$  from the normal direction (ND) in the TD–ND plane, while the maximum of prismatic planes was appeared at inclination angle of  $40\text{--}50^\circ$  in the TD–ED plane. These texture components mostly place the magnesium crystal inclined to

177 the ABE axis. The post-ABE texture may be discussed relying on the presence of favorable  
 178 initial basal texture, where the c-axis is mostly oriented by 40–50° from the shear direction  
 179 during ABE [32]. With the application of subsequent passes, there is a tendency of the basal  
 180 plane lying parallel to TD, while the orientation of prismatic planes shows a random distribution  
 181 around ND. Accordingly, after four passes it is evident that the most noticeable texture includes  
 182 fiber basal texture oriented almost parallel to the transverse direction, and <10–10>  
 183 perpendicular to the ED.  
 184



**Fig. 4. Typical grain size distribution of the experimental alloy processed by (a) one pass, (b) two passes and (c) six passes at 553 K (280 °C) .**

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**b)**

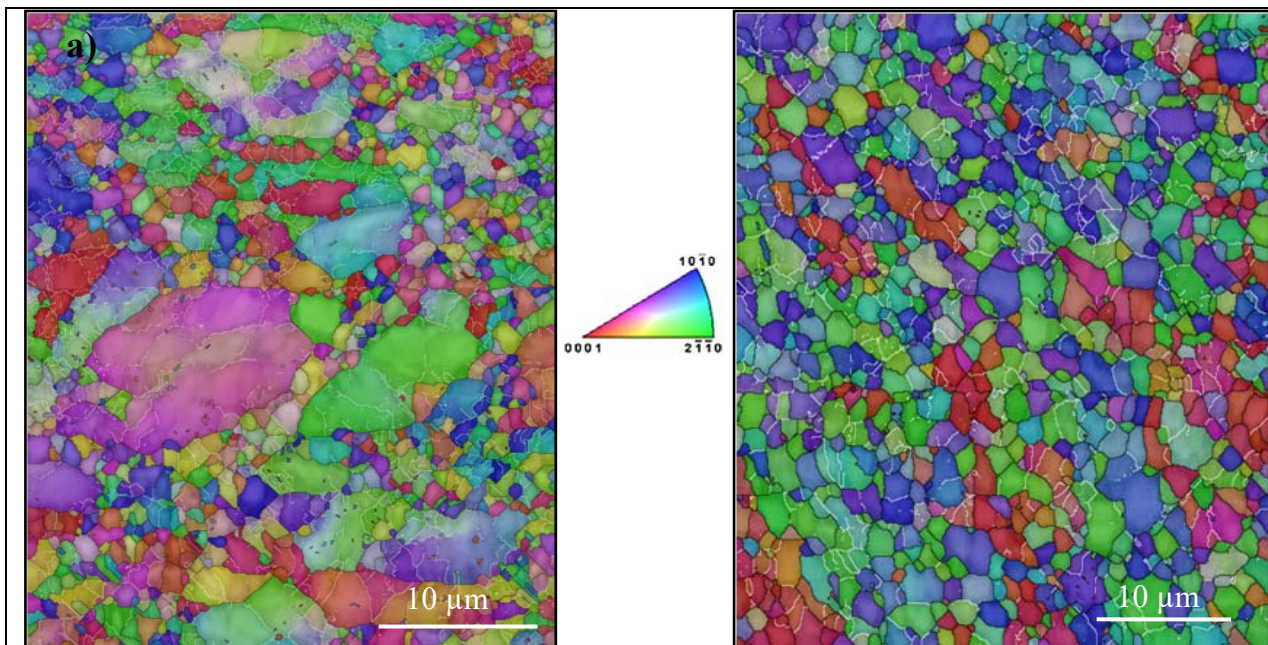


Fig. 5. Inverse pole figures of the material deformed up to a) one pass, b) four passes. **White and black lines denoted boundaries with misorientation below and above 15°, respectively.**

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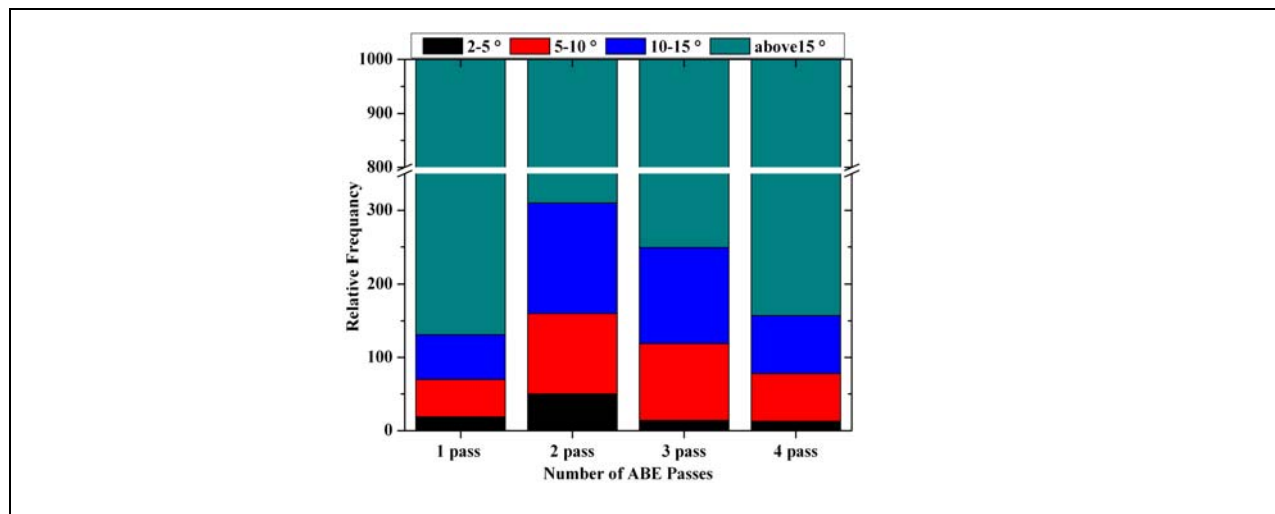
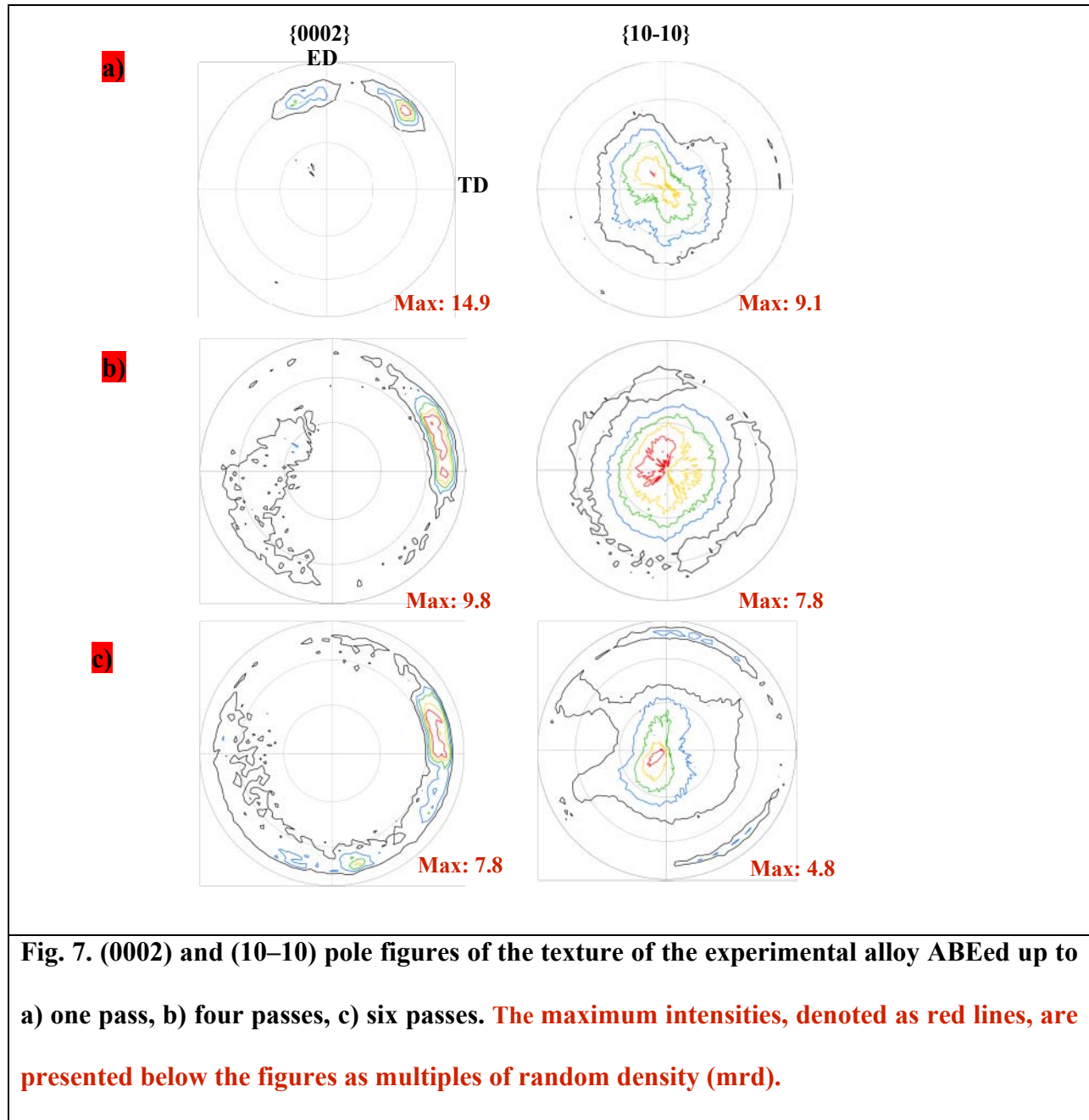


Fig. 6. Cumulative frequency of boundaries with different misorientation angle in AZ31 alloy deformed to various number of ABE passes.



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Fig. 8(a) shows tensile stress vs. strain for the coarse-grain as-received and UFG as-processed alloys at room temperature. However, one can readily notice that the mechanical properties were significantly altered after ABE deformation. The data of yield stress (YS), ultimate tensile strength (UTS) and fracture strain are summarized in Fig. 8b. The yield stress slightly drops after the first pass, and then increases as subsequent passes are applied. The UTS

194 also decreased after the first pass, while it continues to ascend with increasing number of passes,  
195 where a UTS of 250 MPa was reached after six ABE passes. The elongation of processed  
196 material gradually increased where an exceptional ductility of about 68 pct was measured after  
197 multiple ABE passes. The correlation between grain size and yield stress based on Hall-Petch  
198 relation was established in Fig. 9. It is obvious that the data belonging to the as-received alloy is out  
199 of line with the ones obtained from processed materials. The enhanced yield strength of the as-  
200 received alloy comparing the plotted Hall-Petch correlation is related to the different deformation  
201 mode dominating at the beginning of plasticity. This will be discussed later in this paper.

202         Considering the miniaturized tensile samples employed in this study, one may expect a  
203 greater post-uniform elongation comparing to standard samples. Gauge length tends to have  
204 significant effects on the post-uniform elongation and the total ductility. The shorter the gage  
205 length the greater the total elongation is expected [26, 34]. Moreover it has been found in the  
206 case of ultrafine grained Cu that thicker specimens tend to be more ductile, too. The thickness  
207 effect is mainly caused by the necking geometry and/or fracture modes [34]. Thus to compare  
208 results with the literature the effect of specimen geometry should be considered. A method for  
209 determination of stable strain before maximum load for the as-processed alloy is using  
210 Considere's construction:

211

$$212 \quad \frac{d\sigma}{d\varepsilon} = \sigma \quad (\text{eq. 1})$$

213 where  $\sigma$  and  $\varepsilon$  refer to true stress and true strain. The variations of work hardening rates were  
214 included as dashed lines in Fig. 8a. The portions beyond the intersection point on the curves  
215 represent post uniform deformation. It is evident that both uniform and non-uniform strains are  
216 improved in the fine-grained processed materials.

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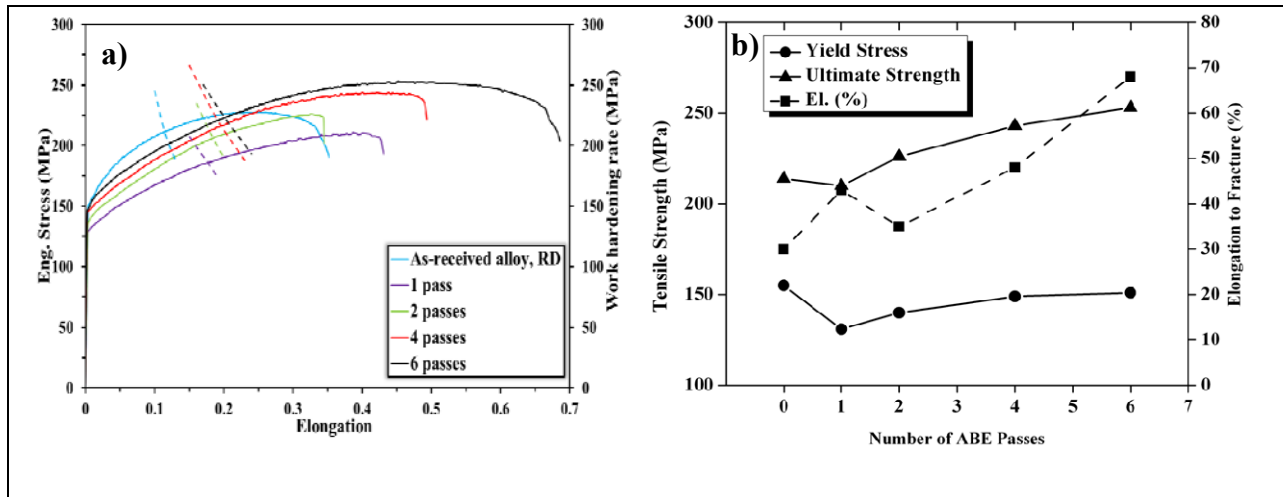


Fig. 8 a) Tensile true stress versus true strain at room temperature for as-received and processed experimental alloy, b) Variation of the yield stress, UTS and fracture strain with the number of ABE passes. The dashed lines are corresponding hardening rates.

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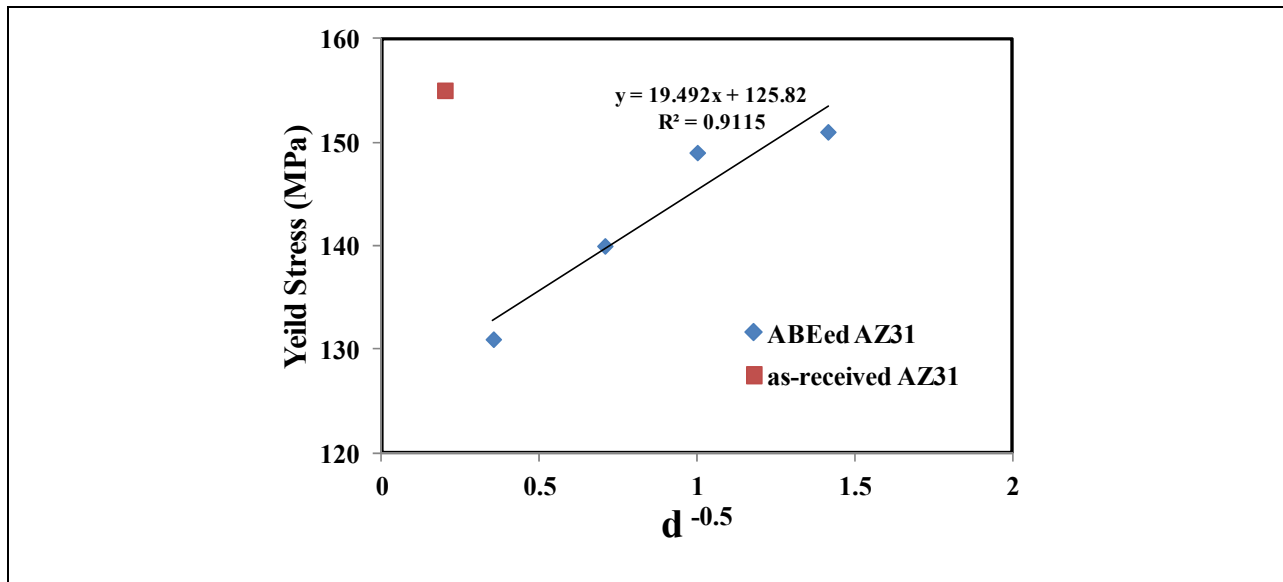
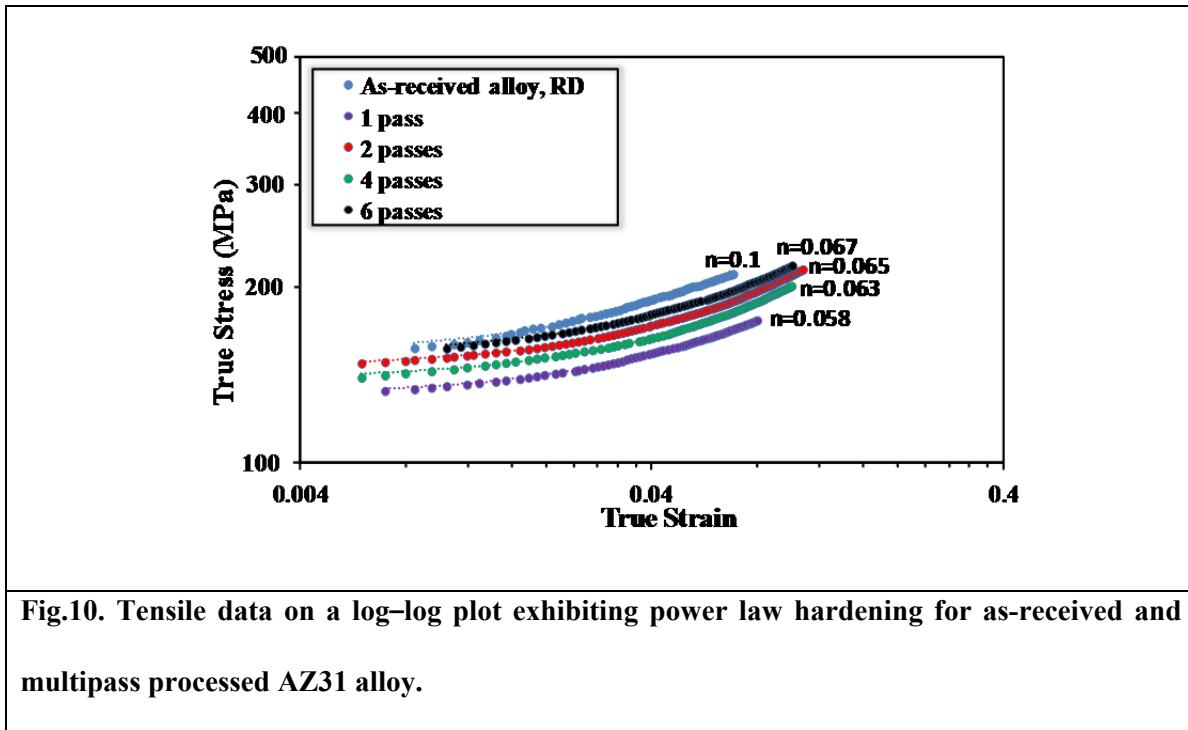


Fig.9. the Hall-Petch correlation of yield stress and grain size for the as-received and processed alloys.

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221 A power law constitutive relation ( $\sigma = K\varepsilon^n$ ) is shown to fit the flow curves with  
222 reasonably accuracy for true-strains up to about 0.15 (Fig. 10). The deviation from the linear  
223 relationship may be related to the changes in the share of different slip systems as the strain  
224 accommodation progressed and thus to the slight change in  $n$  value. The results implied that the  
225 hardening exponent ( $n$ ) was slightly decreased after ABE processing, which is connected to the  
226 grain refinement effect. However, opposed to the grain refining effect, the evolution of texture  
227 caused a decrease in the yield stress as well as a slight increase in uniform elongations, as will be  
228 further discussed in the following paragraphs. A large post uniform elongation is promoted by  
229 preventing localization of the deformation as a sharp neck. This may be introduced due to the  
230 increased rate of thermally activated processes such as dislocation climb and GBS [35]. The  
231 occurrence of GBS at ambient temperature was associated to a large strain rate sensitivity at the  
232 vicinity of the grain boundary [36]. Since the volume of grain boundaries increases with grain  
233 refinement, ultrafine and nano-grained magnesium may exhibit an extended non-uniform  
234 deformation. It is worth noting that total elongation was relatively deteriorated after the second  
235 pass (see Fig. 8). This observation may be attributed to the increased frequency of LABs after the  
236 second pass. It was previously reported that the mechanism of strain propagation through  
237 boundaries with misorientations of  $<35^\circ$  may limit strain accommodation by this mechanism  
238 [37].



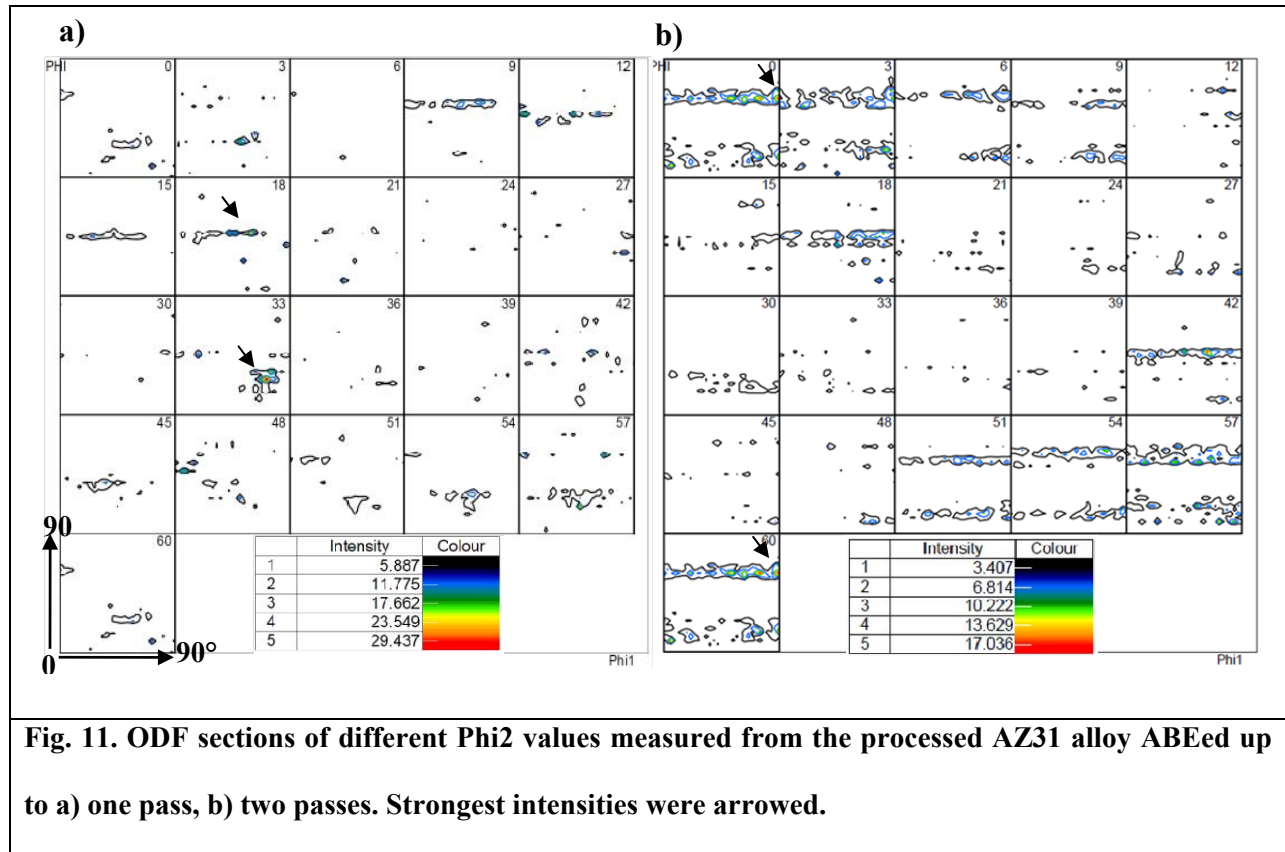


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240 One way to quantify the texture data is to use the crystallite orientation distribution  
 241 function (ODF), which essentially describes the frequency of occurrence of particular  
 242 orientations in a three-dimensional (Euler) orientation space. To identify the degree to which  
 243 twinning and/or slip systems contribute during tensile deformation of ABEed materials, the  
 244 ODFs were obtained from x-ray diffraction texture data on the basis of spherical harmonics with  
 245 positivity correction according to Dahms and Bunge [38]. ODFs are typically depicted for one  
 246 and two-pass processed materials in Fig. 11. Accordingly, the crystallographic plane and  
 247 direction which lie, in most of grains, parallel to the extrusion direction of ABEed samples (and  
 248 thus parallel to tension axis) could be defined. The corresponding Euler angles, planes and  
 249 directions were given in Table 1. The defined directions correspond to the deformation axis of  
 250 the tension samples. The HCP crystal lattice of magnesium features the six equivalent twinning  
 251 planes, the Schmid factors of which can be calculated numerically for any possible orientation in  
 252 the textured polycrystalline alloy. For the twinning systems the variant with maximum Schmid

253 factor and for the slip systems the average values of Schmid factors were considered as effective  
254 Schmid factors. For the samples with two intensity peaks the values proposed by the two were  
255 averaged out.

256 In the present paper, the system with the highest Schmid factor is supposed to become  
257 activated. For the as-received material most grains have basal poles aligned almost perpendicular  
258 to the RD, maintaining a  $\langle 1-210 \rangle$  pole peak parallel to the tension direction. As  $\{10-12\}$  -tensile  
259 twinning creates extension along the c-direction it cannot operate for tensile loads in the basal  
260 plane, though it possesses a proper **absolute** values of Schmid factor for activation. **The latter**  
261 **refers to a case of negative Schmid factor for twinning system.** So, the texture of initial rolled  
262 material does not favor the tensile twinning during RD tension. Also, as the Schmid factor is not  
263 appropriate to activate basal slip, the constraint of the limited number of independent slip  
264 systems cause a high yield stress. The flow stress increases to reach critical shear stress for  
265 prismatic slips, which are favored with Schmid factor of 0.37. Moreover,  $c+a$  slip having  
266 Schmid factor of 0.17 may also contribute at higher strain to contract the material along c-axis  
267 [39].The strain hardening behavior observed along the RD is consistent with the suppression of  
268 twins, where monotonic exhausted hardening rate was recorded. The latter hardening behavior  
269 led to a “convex” shape of flow curve with respect to the upper end of the stress axis (see Fig. 8).



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271 As the material processed through single-pass ABE, the Schmid factor calculated for the  
 272 activation of twinning is lowered but yet considerable, according to the Table 1. However, it  
 273 should be noted that due to the grain refinement introduced during ABE, the critical stress  
 274 required for twinning significantly increases. Thus, twinning may hardly be involved during  
 275 tensile tests of ABEed materials. Previous works has denoted that the occurrence of twinning is  
 276 stopped in AZ31 alloy at a grain size of 3  $\mu\text{m}$  [26, 40] and 3.4  $\mu\text{m}$  [41].

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278 The main texture components of the processed alloy have the c-axis of most of the grains  
 279 distributed inclined to the ABExtrusion direction, though it tends to rotate toward transverse  
 280 direction with increasing number of passes (Fig. 7). This orientation is commonly described as a  
 281 *soft* orientation that is favorable for easy slip modes. According to Table 1, the activation of

282 basal slip system is favored after ABE processing. Thus, easy glide on basal planes may be  
283 considered as the primary deformation mechanism. The critical resolved stress for activation of  
284 basal slip is much less than prismatic one [42]. This fact may help to explain the drop in the yield  
285 stress govern the occurrence of slip during tensile loading of ABEed materials. These evidences  
286 implied that the texture softening resulted from ABE texture overcome the hardening effect  
287 generated by grain refinement. The fraction of dynamic recrystallization increased with applying  
288 consecutive passes, and the mean grain size of the ABEed alloy markedly lowered. This may  
289 harden the material by impeding basal slips and grain boundary strengthening effect. Moreover,  
290 the latter can be amplified by weakening of the basal texture intensity, which limits, in turn, the  
291 share of easy glide on basal plane during deformation. As the tensile deformation of ABEed  
292 samples progresses and hardening occurs on basal planes, prismatic slip can be contributed in  
293 deformation. According to the initial texture in tension samples a  $\langle 1-120 \rangle$  prismatic slip may  
294 accommodate the plastic strain through contraction in the thickness direction. Moreover,  $\langle 1-$   
295  $100 \rangle$  double slip (i.e. concurrent activation of two prismatic slip systems; e.g.  $(0\ 1\ -10)$   $[2-1-10]$   
296 and  $(1-1\ 0\ 0)$   $[1\ 1-2\ 0]$ ) may incorporate in the elongation of the sample. Detailed discussion of  
297 double prismatic slip in textured magnesium can be found in work published by Saxl et. al [43].  
298 Thus, prismatic slip as the major deformation mechanism render the shape change favored  
299 during tension test. A significant role of double prismatic slip in enhancing the ductility of  
300 magnesium alloys were previously reported by Kainer et. al. [44] and Al-Samman et. al. [8].  
301 Accordingly, the operation of prismatic slip results in the orientation of individual grains  
302 remaining virtually unchanged during deformation [8]. Moreover, GBS also occurs at room  
303 temperature up to 8 pct of total strain, enhanced by plastic anisotropy as well as by the increasing  
304 number of grain-boundary dislocations [39]. GBS at room temperature is related to slip induced

305 inhomogeneous plastic deformation of the ultrafine/nano grained processed material. The  
306 positive role of GBS in enhancing ductility should be associated with the activity of non-basal  
307 slips [6, 39]. In present work, the desired orientation condition for operation of prismatic slip  
308 systems and thereby concentrated stresses at the grain boundaries caused by dislocation slip  
309 invoke the occurrence of GBS. This mechanism is considered to give rise to considerable  
310 ductility improvement in ultrafine/nano grained ABEed magnesium. As the number of passes  
311 increased, further reducing of the mean grain size as well as improving the Schmid factor for  
312 operation of prismatic slip resulted in a promoted boundary sliding associating with a significant  
313 ductility of 68 pct. Somekawa and Mukai [45] showed a superplastic elongation of 230 pct at  
314 room temperature and strain rate  $10^{-5} \text{ s}^{-1}$  in pure Mg having a mean grain size of  $1.2 \mu\text{m}$ . More  
315 recently, excellent ductilities were recorded in pure magnesium with even finer grain structures  
316 including an elongation of 360 pct in a sample with a grain size of 320 nm tested at room  
317 temperature at a low strain rate of  $10^{-5} \text{ s}^{-1}$  [19]. The latter improved ductilities were also  
318 attributed to an enhanced contribution from grain boundary sliding to the overall deformation  
319 mechanism.

320

## 321 **Summary**

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323 Ultrafine/nano grained microstructure was produced in AZ31 wrought magnesium alloy  
324 through multipass ABE processing. A texture involving major texture component inclined to the  
325 deformation axis was developed. The promotion of easy basal slip due to the texture changes  
326 counteract the strengthening effect of grain refinement, where the yield stress of the experimental  
327 material reduced after processing. The contribution of different twinning and slip system during  
328 room temperature tension of ABEed alloys was discussed relying on the Schmid factor

329 calculated by considering the obtained texture. Both uniform and post uniform elongations of the  
330 material were improved after processing. The occurrence of GBS associated with the operation  
331 of prismatic slip result in a significant post uniform elongation.

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### 333 **References**

334 [1] B. Mordike, T. Ebert: *Mater. Sci. Eng. A*, 2001, 302, 37-45.

335 [2] T. Al-Samman, G. Gottstein: *Mater. Sci. Eng.: A*, 2008, 488, 406-414.

336 [3] H. Yan, R. Chen, E. Han: *Mater. Sci. Eng. A*, 2010, 527, 3317-3322.

337 [4] S. Agnew, J. Horton, T. Lillo, D. Brown: *Scr. Mater.*, 2004, 50, 377-381.

338 [5] J. Del Valle, F. Carreno, O. Ruano, *Acta Mater.*, 2006, 54, 4247-4259.

339 [6] J. Koike, T. Kobayashi, T. Mukai, H. Watanabe, M. Suzuki, K. Maruyama, K. Higashi, *Acta*  
340 *Mater.*, 2003, 51, 2055-2065.

341 [7] S. Agnew, P. Mehrotra, T. Lillo, G. Stoica, P. Liaw: *Mater. Sci. Eng. A*, 2005, 408, 72-78.

342 [8] T. Al-Samman, X. Li, S.G. Chowdhury: *Mater. Sci. Eng. A*, 2010, 527, 3450-3463.

343 [9] S. Biswas, S. Singh Dhinwal, S. Suwas: *Acta Mater.*, 2010, 58, 3247-3261.

344 [10] S. Fatemi-Varzaneh, A. Zarei-Hanzaki: *Mater. Sci. Eng. A*, 2009, 504, 104-106.

345 [11] S. Fatemi-Varzaneh, A. Zarei-Hanzaki, J. Cabrera, P. Calvillo: *Materials Chemistry and*  
346 *Physics*, 2015, 149, 339-343.

347 [12] H. Wang, P. Wu, J. Wang: *Comput. Mater. Sci.*, 2015, 96, 214-218.

348 [13] J. Wang, D. Zhang, Y. Li, Z. Xiao, J. Fouse, X. Yang: *Mater. Des.*, 2015, 86, 526-535.

349 [14] H. Fan, S. Aubry, A. Arsenlis, J.A. El-Awady: *Scr. Mater.*, 2015, 97, 25-28.

350 [15] M. Quadir, M. Ferry, O. Al-Buhamad, P. Munroe: *Acta Mater.* 2009, 57, 29-40.

351 [16] J. Del Valle, P. Rey, D. Gesto, D. Verdera, J.A. Jiménez, O.A. Ruano: *Mater. Sci. Eng. A*,  
352 2015, 628, 198-206.

- 353 [17] M. Gzyl, A. Rosochowski, S. Boczkal, L. Olejnik: *Mater. Sci. Eng. A*, 2015, 638, 20-29.
- 354 [18] S. Biswas, S. Suwas: *Scr. Mater.*, 2012, 66, 89-92.
- 355 [19] R.B. Figueiredo, S. Sabbaghianrad, A. Giwa, J.R. Greer, T.G. Langdon: *Acta Mater.*, 2017,  
356 122, 322-331.
- 357 [20] W. Yuan, S. Panigrahi, J.Q. Su, R. Mishra: *Scr. Mater.*, 2011,
- 358 [21] J. Xing, X. Yang, H. Miura, T. Sakai: *Mater. Trans.*, 2008, 49, 69-75.
- 359 [22] Z. Zuberova, Y. Estrin, T. Lamark, M. Janecek, R. Hellmig, M. Krieger, *J. Mater. Proc.*  
360 *Tech.*, 2007, 184: 294-299.
- 361 [23] W. Kim, C. An, Y. Kim, S. Hong: *Scr. Mater.*, 2002, 47, 39-44.
- 362 [24] H. Miura, G. Yu, X. Yang: *Mater. Sci. Eng. A*, 2011,
- 363 [25] Y. Wang, E. Ma, *Acta Mater.*: 2004, 52, 1699-1709.
- 364 [26] Q. Yang, A. Ghosh, *Acta Mater.*, 2006, 54, 5159-5170.
- 365 [27] N. Haghdadi, A. Zarei-Hanzaki, D. Abou-Ras: *Mater. Sci. Eng. A*, 2013, 584, 73-81.
- 366 [28] B. Bazaz, A. Zarei-Hanzaki, S. Fatemi-Varzaneh: *Mater. Sci. Eng. A*, 2013, 559, 595-600.
- 367 [29] S. Fatemi-Varzaneh, A. Zarei-Hanzaki: *Mater. Sci. Eng. A*, 2011, 528, 1334-1339.
- 368 [30] S. Fatemi-Varzaneh, A. Zarei-Hanzaki, M. Naderi: A.A. Roostaei, *J. Allo. Comp.*, 2010,  
369 507, 207-214.
- 370 [31] S. Fatemi-Varzaneh, A. Zarei-Hanzaki, J. Cabrera: *J. Allo. Comp.*, 2011, 509, 3806-3810.
- 371 [32] S. Fatemi-Varzaneh, A. Zarei-Hanzaki, H. Paul: *Mater. Charac.*, 2014, 87, 27-35.
- 372 [33] J.W. Cahn, Y. Mishin, A. Suzuki, *Acta Mater.*: 2006, 54, 4953-4975.
- 373 [34] Y. Zhao, Y. Guo, Q. Wei, A. Dangelewicz, C. Xu, Y. Zhu, T. Langdon, Y. Zhou, E.  
374 Lavernia: *Scr. Mater.*, 2008, 59, 627-630.

375 [35] W.F. Hosford, R.M. Caddell, Metal forming: mechanics and metallurgy, Cambridge  
376 University Press, 2011.

377 [36] H. Somekawa, T. Mukai: *Phil. Mag. Lett.*, 2010, 90, 883-890.

378 [37] C. Cepeda-Jiménez, J. Molina-Aldareguia, M. Pérez-Prado: *Acta Mater.*, 2015, 84, 443-456.

379 [38] M. Dahms, H.J. Bunge: *J. App. Crys.*, 1989, 22, 439-447.

380 [39] J. Koike: *Metall. Mater. Trans. A*, 2005, 36, 1689-1696.

381 [40] R. Lapovok, P. Thomson, R. Cottam, Y. Estrin: *J. Mater. Sci.*, 2005, 40, 1699-1708.

382 [41] S. Yin, C. Wang, Y. Diao, S. Wu, S. Li: *J. Mater. Sci. Tech.*, 2011, 27, 29-34.

383 [42] S. Agnew, Ö. Duygulu: *Int. J. Plast.*, 2005, 21, 1161-1193.

384 [43] I. Saxl, I. Haslingerová, *J. Phys., B*, 1974, 24, 1351–1361.

385 [44] S. Kleiner, P. Uggowitzer: *Mater. Sci. Eng. A*, 2004, 379, 258-263.

386 [45] H. Somekawa, T. Mukai: *Metall. Mater. Trans. A*, 2015, 46, 894-902.

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**Table 1. Absolute values of Schmid factors calculated for twinning and slip systems in the processed AZ31 alloy assuming the axis of ABEed sample as deformation axis. For the twinning systems the variant with maximum Schmid factor and for the slip systems the average values of Schmid factors were considered as effective Schmid factors. For the samples with two intensity peaks the values proposed by the two were averaged out.**

Pass Number	$g\{\phi_1, \phi, \phi_2\}$	Deformation axis	twinning	max.	ave.	basal slip	average	prismatic slip	average	pyramidal slip	average
0	{81.,0,12}	$\cong (0001)$ $\langle 1\bar{2}10 \rangle$	0.24, 0.42, 0.42	0.42	0.42	0.00, 0.00, 0.00	0.00	0.00, 0.45, 0.27	0.37	0.22, 0.07, 0.22	.017
	{81,0,57}		0.00, 0.24, 0.00								
1	{72,63,33}	$\cong (01\bar{1}1)$ $\langle 1\bar{2}11 \rangle$	0.26, 0.28, 0.24	0.21	0.21	0.07, 0.23, 0.00	0.15	0.02, 0.07, 0.00	0.03	0.03, 0.13, 0.01	0.06
			0.19, 0.28, 0.21								
	{60,45,18}	$\cong (01\bar{1}1)$ $\langle 1\bar{1}01 \rangle$	0.10, 0.21, 0.21	0.21		0.30, 0.34, 0.07	0.22	0.00, 0.23, 0.07	0.10	0.04, 0.16, 0.06	0.09
			0.14, 0.14, 0.03								
2	{90,30,0}	$\cong (\bar{1}2\bar{1}1)$ $\langle 1\bar{2}11 \rangle$	0.12, 0.19, 0.00	0.19	0.28	0.15, 0.43, 0.22	0.27	0.10, 0.32, 0.00	0.14	0.00, 0.10, 0.16	0.09
	{90,30,60}	$\cong (11\bar{2}1)$ $\langle \bar{1}\bar{1}21 \rangle$	0.18, 0.19, 0.07	0.37		0.27, 0.33, 0.42	0.34	0.01, 0.23, 0.17	0.14	0.04, 0.16, 0.04	0.08
			0.34, 0.04, 0.37								
4	{69.,45,18}	$\cong (01\bar{1}1)$ $\langle 1\bar{1}01 \rangle$	0.22, 0.11, 0.39	0.39	0.39	0.24, 0.35, 0.08	0.22	0.30, 0.04, 0.14	0.18	0.07, 0.15, 0.00	0.07
6	{0.,60,30}	$\cong (01\bar{1}1)$ $\langle 2\bar{1}10 \rangle$	0.27, 0.00, 0.27	0.41	0.45	0.00, 0.00, 0.00	0.00	0.04, 0.28, 0.22	0.21	0.07, 0.07, 0.26	0.13
	{90,60,18}	$\cong (01\bar{1}1)$ $\langle 0\bar{1}11 \rangle$	0.18, 0.27, 0.28	0.49		0.03, 0.21, 0.12	0.12	0.00, 0.04, 0.00	0.02	0.06, 0.12, 0.00	0.09
			0.23, 0.37, 0.49								

## **Figure captions**

**Fig. 1. a) Initial microstructure, b) measured pole figures of the experimental as-rolled AZ31 alloy.**

**Fig. 2. a) Schematic illustration of geometry and orientation of tension specimen used in mechanical testing, b) tension machine for testing miniaturized samples.**

**Fig. 3. Microstructure of the experimental alloy deformed up to a) one, b) two, c) five passes at 553 K (280°C),d) ultrafine/nano grains formed during ABE processing**

**Fig. 4. Typical grain size distribution of the experimental alloy processed by (a) one pass, (b) two passes and (c) six passes at 553 k (280 °C).**

**Fig. 5. Inverse pole figures of the material deformed up to a) one pass, b) four passes. White and black lines denoted boundaries with misorientation below and above 15°, respectively.**

**Fig. 6. Cumulative frequency of boundaries with different misorientation angle in AZ31 alloy deformed to various number of ABE passes.**

**Fig. 7. (0002) and (10–10) pole figures of the texture of the experimental alloy ABEed up to a) one pass, b) four passes, c) six passes. The maximum intensities, denoted as red lines, are presented below the figures as multiples of random density (mrd).**

**Fig. 8 a) Tensile true stress versus true strain at room temperature for as-received and processed experimental alloy, b) Variation of the yield stress, UTS and fracture strain with the number of ABE passes. The dashed lines are corresponding hardening rates.**

**Fig.9. the Hall-Petch correlation of yield stress and grain size for the as-received and processed alloys.**

**Fig.10. Tensile data on a log–log plot exhibiting power law hardening for as-received and multipass processed AZ31 alloy.**

**Fig. 11. ODF sections of different  $\Phi^2$  values measured from the processed AZ31 alloy ABEed up to a) one pass, b) two passes. Strongest intensities were arrowed.**