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Effect of Microstructural Factors on Tensile Properties of an ECAE-Processed AZ31 Magnesium Alloy*1

Yu Yoshida*2, Lawrence Cisar*2, Shigeharu Kamado and Yo Kojima

Department of Mechanical Engineering, Nagaoka University of Technology, Nagaoka 940-2188, Japan

Mg–3%Al–1%Zn (AZ31) alloy was subjected to ECAE (Equal Channel Angular Extrusion) processing under various processing conditions. Then tensile tests were carried out at room temperature to investigate the relationship between tensile properties and microstructural parameters that include grain size and the texture generated by ECAE processing. In 4-pass ECAE specimens processed at 523 K, tensile ductility is improved as a result of easy basal slip during tensile test along the extrusion direction, because such specimens have textures in which the basal plane is inclined at 45° to the extrusion direction. On the other hand, in the specimens processed at 573 K, 0.2% proof stress is higher than those of specimens processed at lower temperatures, but elongation is smaller. This is because of difficult basal slip caused by the textures in which the basal plane is oriented parallel to the extrusion direction. However, 8-pass specimens processed at 473 K and subsequently annealed, which have similar textures but different grain sizes (d), exhibit clear grain size dependencies of 0.2% proof stress ($\sigma_{0.2}$) according to Hall-Petch relationship; $\sigma_{0.2} = 30 + 0.17d^{-1/2}$. Therefore, crystallographic orientation has a profound effect on the tensile properties of AZ31 alloy, and grain size has a little effect.

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Keywords: *equal channel angular extrusion process, magnesium alloy, tensile properties, texture, grain size dependence*

1. Introduction

Although magnesium alloys are expected to be useful materials for minimizing global environmental problems, wider applications of the alloys have not been achieved due to their inferior cold workability. Generally, it has been said that the less cold workability of magnesium results from its limited slip system at room temperature. Recently, however, there are some instances[1–4] where the ductility is improved in grain-refined magnesium alloys even at room temperature. This suggests that the poor ductility of magnesium relates to microstructural conditions, and not necessarily to intrinsic nature of the material.

There is a large number of reports[4–12] about the correlation between grain size and mechanical properties of magnesium alloys based on Hall-Petch relation. In magnesium, the critical resolved shear stress (CRSS) for non-basal slip is about one hundred times as large as that for basal slip.[13] This difference of the CRSS gives rise to anisotropy of mechanical properties resulting from the slip system. Consequently, when the mechanical properties of wrought magnesium alloys that have strong crystallographic texture in their structures are investigated, considering the crystallographic orientation in addition to the grain size is important.[6,7]

In cold rolled magnesium alloys, the basal plane is oriented parallel to the rolling direction, that is, the so-called basal texture is formed.[14,15] The basal texture is also generated in hot rolled magnesium alloys.[7,15,16] Furthermore, in extruded magnesium alloys, the texture in which the basal plane is oriented parallel to the extrusion direction is formed.[6,12,16,17] If these materials were deformed along the working direction, the ductility is expected to be small because shear force does not operate on the basal plane due to small Schmid factor that is nearly 0. Therefore, their workability will also be small when secondary working is carried out at room temperature using the rolled or extruded materials.

In a previous study,[18] ECAE processing[19,20] was applied to Mg–Al–Zn alloys as a method not only for grain refinement but also for texture control. As a result, a peculiar texture in which the basal plane is inclined at 45° to the extrusion direction was formed, and therefore, the ductility was improved significantly. However, the relationship between ECAE processing conditions and the formed microstructures including texture, as well as the effect of the microstructure on tensile properties of the ECAE-processed specimen have not been clarified yet.

In the present study, extruded AZ31 magnesium alloy was subjected to ECAE processing. Then the effect of ECAE processing conditions, such as the process temperature and repetitive number of ECAE process, on grain size and texture formation was examined. Subsequently, the ECAE-processed specimens were subjected to tensile tests. Then the correlation between tensile properties and microstructural parameters, such as grain size and texture are investigated. Furthermore, the effect of microstuructural changes with annealing on tensile properties of ECAE-processed specimens is also investigated.

2. Experimental Procedure

Commercial AZ31 extruded rods with 19 mm diameter were obtained for the investigations carried out in the present study. The chemical composition of the alloy is shown in Table 1. Figure 1 shows a schematic illustration of the ECAE

Table 1 Chemical composition of AZ31 alloy used for present study (mass%).

Alloy	Al	Zn	Mn	Fe	Si	Cu	Ni	Mg
AZ31	3.26	0.92	0.43	0.004	0.001	0.000	0.001	bal.

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*2Graduate Student, Nagaoka University of Technology.

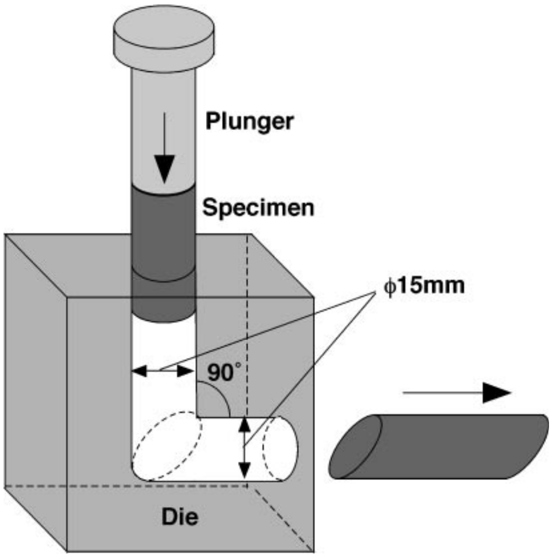

Fig. 1 Schematic illustration of ECAE process.

process used for the present study. During the ECAE processing, the specimen is inserted into the upper part of the die and pressed. Then the specimen is subjected to shear force at the intersecting point of two equal channels. The ECAE die used for the present study has two equal channels of 15 mm diameter. The intersecting angle between the two channels is 90° and the angle of the outer arc at the intersection is 60°. Using this ECAE die, an equivalent strain of 94% can be introduced.[21)]

The extruded bars were machined to cylindrical specimens with a diameter of 15 mm and a length of 80 mm for ECAE processing. Table 2 shows the ECAE processing conditions. The ECAE processing was carried out at 473, 523 and 573 K, respectively, with constant extrusion rate of 20 mm/min. The processing was repeated 2, 4 and 8 times with the specimens rotated at the angles shown in Table 2 around the specimens longitudinal axis. Furthermore, 8-pass ECAE specimens processed at 473 K were subjected to isochronal annealing at 448–673 K for 1 h and at 673–773 K for 4 h in order to investigate the correlation between grain size and tensile properties.

Optical microscope observations of all specimens were performed, and the grain size was simultaneously evaluated by image processing analysis. Furthermore, in the annealed specimens, Vickers hardness test was carried out and the change in hardness with annealing was investigated. Tensile tests were carried out at room temperature under a strain rate of $8.33 \times 10^{-4}\,s^{-1}$. The ECAE-processed samples were machined to JIS14A tensile specimens having a gage dimension of 4 mm diameter and 20 mm length, with their central axes remaining the same as before ECAE processing. After tensile test, the microstructures near the fractured surfaces were observed using optical microscope.

In order to analyze the textures generated by plastic working, (0002) pole figures were drawn by Schulz reflection method[22)] using X-ray diffractometer. The measurements were performed using CuKα (wave length $\lambda = 0.15406$ nm) radiation at 40 kV and 200 mA with the sample tilt angle ranging from 0–70°.

Table 2 ECAE processing conditions.

Number of processing	2, 4			8
Specimen rotation (deg)	2nd: 180 3rd: 90 4th: 180			90
Working temperature (K)	473	523	573	473
Extrusion rate (mm/min)	20			
Annealing condition	—			448–673 K-1 h 673–773 K-4 h

3. Results and Discussion

3.1 Microstructures and tensile properties of as-ECAE specimens

3.1.1 Microstructures of as-ECAE specimens

Figure 2 shows microstructures of as-received specimen, 2 and 4-pass ECAE processed specimens (hereafter referred to as 2-pass and 4-pass specimens, respectively). The microstructures of all ECAE specimens consist of many undeformed equiaxed grains. This suggests that dynamic recrystallization occurs during ECAE processing. Average grain sizes increase with increasing processing temperature in both 2 and 4-pass specimens. However, the average grain sizes decrease with increasing number of processing at each temperature. In the specimen processed at 473 K, although very fine grains of about 1 µm are observed, some coarse

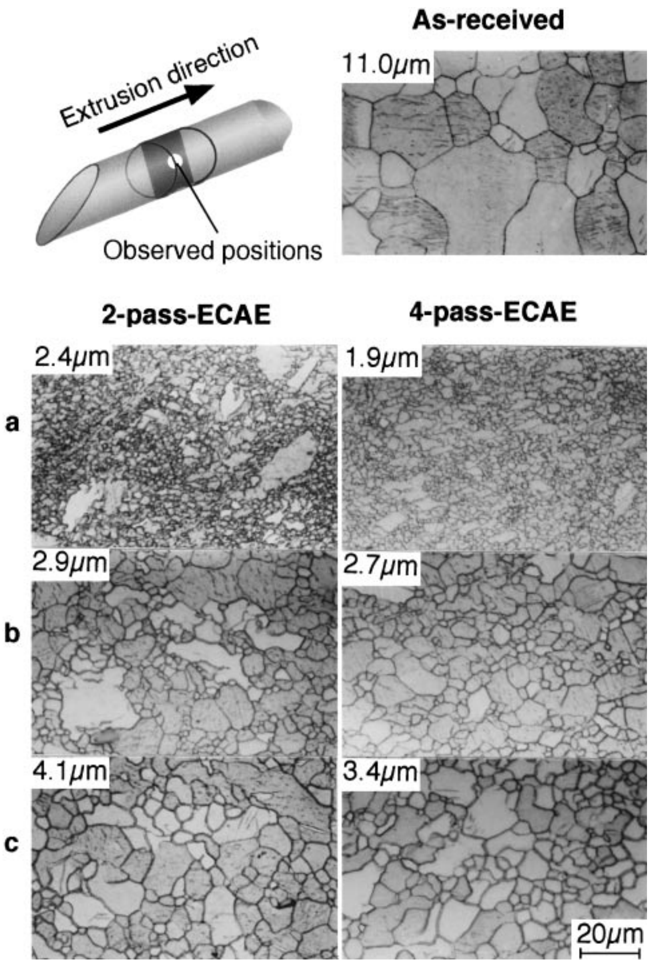

Fig. 2 Microstructures of as-received and ECAE specimens processed at (a) 473 K, (b) 523 K and (c) 573 K. Grain sizes are indicated in the figure.

unrecrystallized grains still remain. This is because the low temperature processing makes both dynamic recrystallization and subsequent grain growth difficult. Furthermore, the homogeneity of the grain sizes is improved by repeating the processing. On the other hand, in the specimens processed at 523 K and 573 K, the variation in grain sizes becomes small. This is as a result of increase in the volume fraction of recrystallized grains generated by dynamic recrystallization during ECAE processing and subsequent grain growth due to relatively high temperature processing.

From the above results, it can be concluded that the processing temperature and the number of repetitive processing have profound effect on the recrystallized grain size and homogeneity of the microstructure, respectively. Furthermore, in high temperature processing, although the grain sizes are large, homogeneous microstructure can be obtained with a small number of processing. For the achievement of fine and homogeneous microstructure, a large number of processing at a relatively low temperature will be required.

3.1.2 Tensile properties

Figure 3 shows stress-strain curves of the as-received specimen and as-ECAE specimens processed under various conditions. Both the tensile strength and the 0.2% proof stress of the 2-pass specimens increase with increasing processing temperature. The stress-strain curve of the 573 K-2-pass specimen is similar to that of the as-received specimen. However, the 523 K-2-pass specimen exhibits an elongation of 25%, which is higher than that of the as-received specimen (19%), while the elongation of the 473 K-2-pass specimen is merely 8%. This value of 8% is much lower than that of the as-received specimen. On the other hand, the elongation of the 4-pass specimens processed at 473 K and 523 K are improved significantly compared to that of the 2-pass specimens processed at those temperatures. Comparing 473 K-4-pass specimen with 523 K-4-pass specimen, the tensile strength and 0.2% proof stress are almost same. However, the elongation of the latter specimen is higher than that of the former specimen. Furthermore, the tensile properties of 573 K-4-pass specimen are almost the same as those of the 573 K-2-pass specimen. However, the 0.2% proof stress of the former is slightly lower than that of the latter.

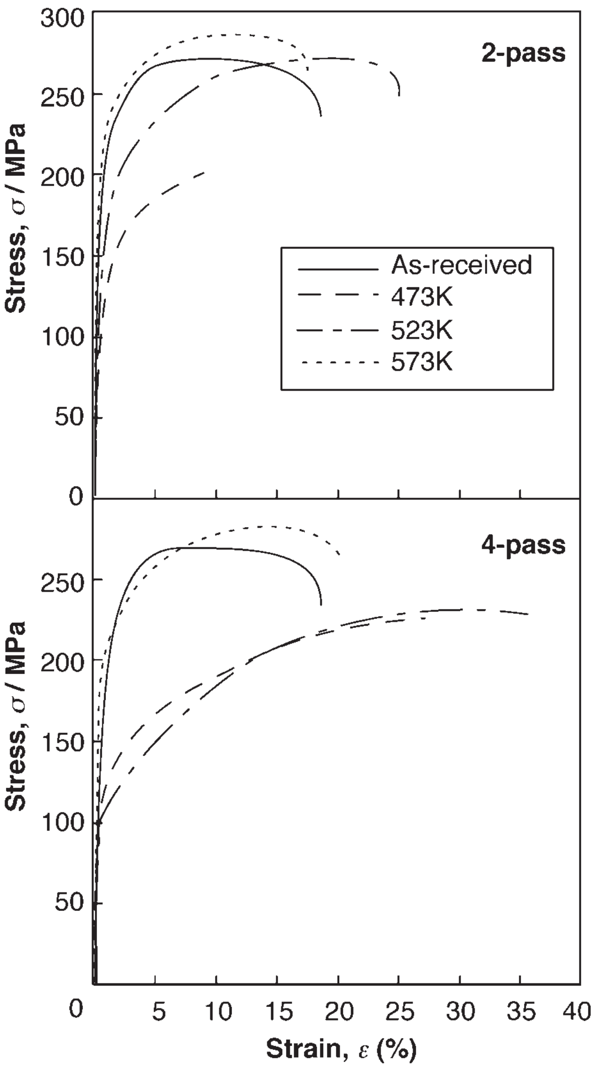

Fig. 3 Stress-strain curves of 2-pass (top) and 4-pass (bottom) ECAE specimens processed at various temperatures.

As described in section 3.1.1, the grain size decreases with decreasing ECAE processing temperature. Generally, in discussing the strength of a material, Hall-Petch relation is often used. However, such grain size dependency of the 0.2% proof stress could not be found from the present results. Therefore, it is inferred that the tensile properties, especially 0.2% proof stress, must have been influenced by the crystallographic texture due to strong anisotropy of the slip systems at room temperature. The relationship between tensile properties and textures will be discussed in details in section 3.1.4.

3.1.3 Microstructures of tensile fractured specimens

Figure 4 shows microstructures near the fractured surfaces of the tensile tested specimens. Some twins are observed in the coarse grains in the specimens processed at 473 K. The existence of twins indicates that the deformability of the coarse grains is lower than that of the fine grains. Consequently, the coarse grains may be responsible for the low elongation of these specimens. In fact, the elongation is enhanced in the 473 K-4-pass specimen that has less coarse grains than the 473 K-2-pass specimen. On the other hand, the microstructures of the specimens processed at 523 K are homogeneous without coarse grains. It is noted that the grains of 523 K-4-pass specimen that exhibits high elongation of 35% are elongated towards the tensile direction. This elongation of the grains suggests that dislocation slip operates actively in the grains and that contributes to the ductility of the specimen. Furthermore, some twins are observed in the 523 K-4-pass specimen, but the twins are uniformly distributed in the microstructure. This result indicates that the deformation is uniform in each grain. Thus, the uniform deformation of the grains may cause improvement of the ductility. A large number of twins are observed in the specimens processed at 573 K. In these specimens, it is considered that twinning also contributes to the whole deformation in addition to dislocation slip. This deformation mechanism is different from that of the specimens processed at 523 K because the grains of the specimens processed at 573 K remain equiaxed.

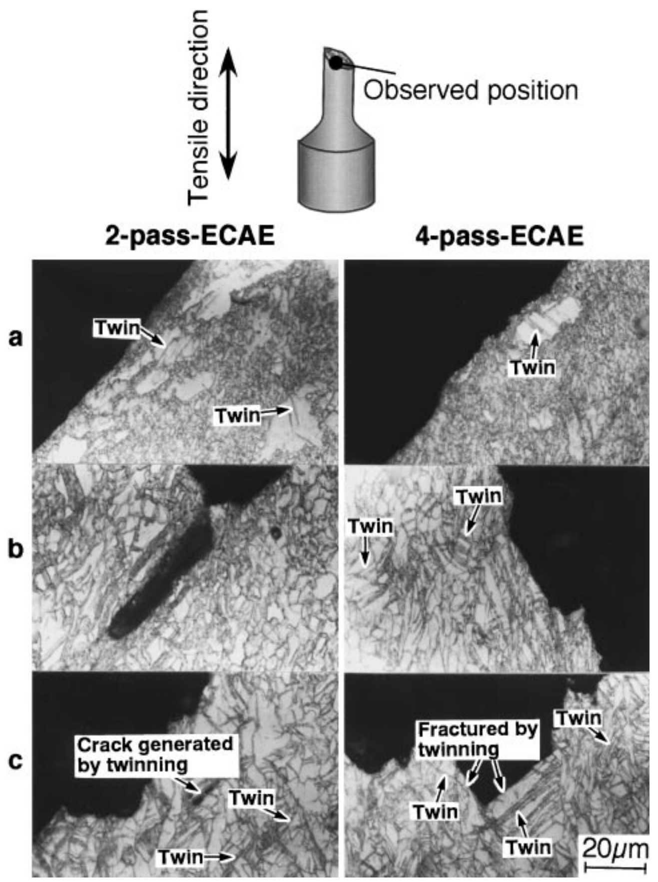

Fig. 4 Microstructures near the fractured surfaces of ECAE specimens processed at (a) 473 K, (b) 523 K and (c) 573 K.

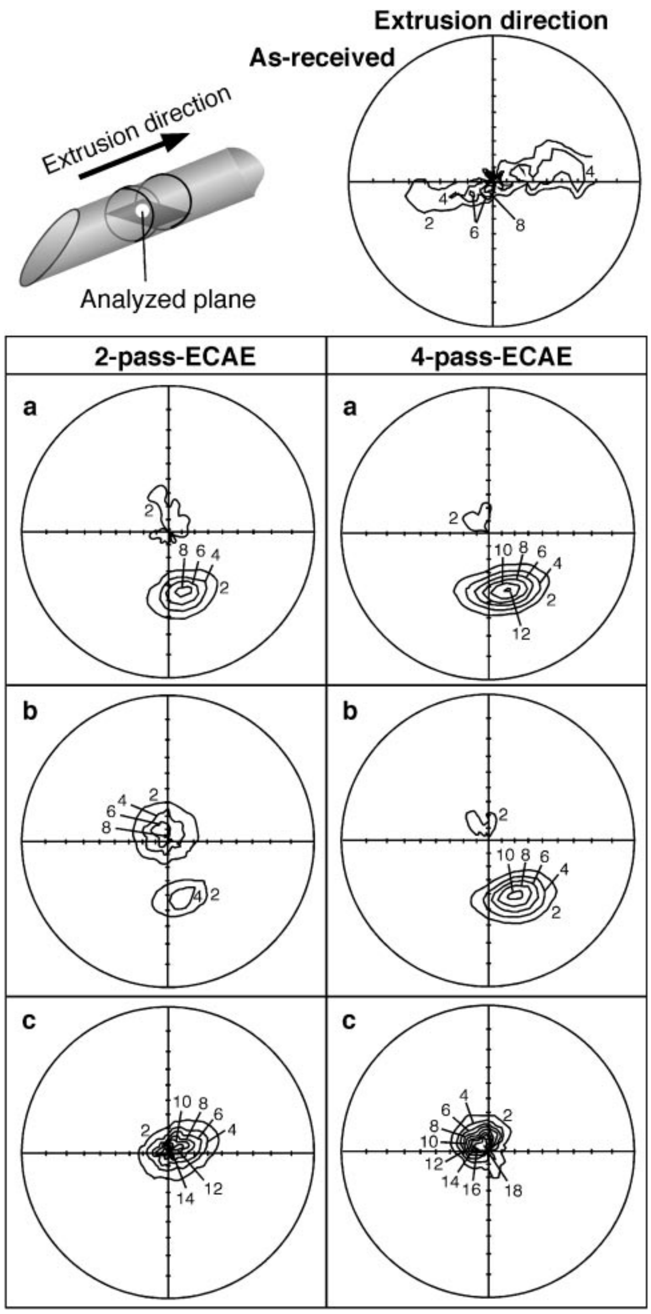

Fig. 5 (0002) pole figures of as-received and ECAE specimens processed at (a) 473 K, (b) 523 K and (c) 573 K.

3.1.4 Texture

Figure 5 shows (0002) pole figures of as-received and as-ECAE specimens. The measurements were carried out on the planes parallel to the extrusion direction.

The basal plane of the as-received specimen is oriented parallel to the extrusion direction. This is the typical texture observed in extruded rods of magnesium alloys.[6,12)]

In the 473 K-2-pass specimen, the texture consists of high intensity basal planes inclined at 45° to the extrusion direction and lower intensity ones parallel to the extrusion direction. The intensity of the former decreases with increasing processing temperature, while that of the latter increases. In the 573 K-2-pass specimen, the basal plane is only oriented parallel to the extrusion direction. The 473 K and 523 K-4-pass specimens have similar textures. In these specimens, the intensity of the basal planes parallel to the extrusion direction decreases compared to 2-pass specimens processed at same temperature. However, the intensity of the inclined basal planes increases. Furthermore, the intensity of the basal plane parallel to the extrusion direction of the 573 K-4-pass specimen is higher than that of the 573 K-2-pass specimen. Thus, repetitive ECAE processing makes the obtained texture strongly exhibit preferred orientation according to the processing temperatures.

The textures generated by ECAE processing can be classified into two types according to the orientations of the basal plane. These orientations are represented schematically in Fig. 6. At a moderately low temperature of about 473 K, the basal plane is inclined at 45° to the extrusion direction along the shear plane during the ECAE processing. On the other hand, at a relatively high temperature of about 573 K, the basal plane is oriented parallel to the extrusion direction. Furthermore, in both texture (a) and (b), two types of orientations that are misaligned at 30° around the c-axis can be expected according to a previous study.[18)] If the specimen having texture (a) is tensioned along the extrusion direction, easy basal slip would occur because maximum shear force occurs on the plane inclined at 45° to the tensile direction. As a result, both the tensile strength and 0.2% proof stress would be remarkably reduced. It is expected that the easy basal slip will also be related to the enhancement of the elongation. In fact, the elongation reaches an extraordinarily high value of 35% in the 523 K-4-pass specimen that has a homogeneous

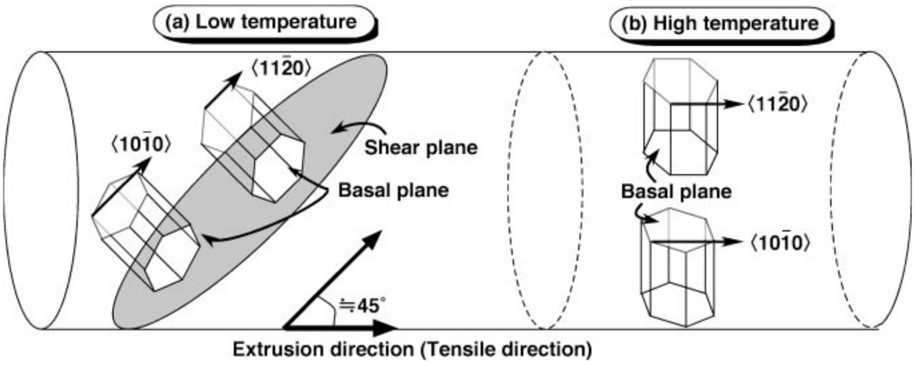

Fig. 6 Schematic illustration of crystallographic orientations formed by ECAE processing.

microstructure. However, it is difficult for the grains to be significantly elongated, as shown in Fig. 4, by only basal slip because the independent slip modes of basal slip are only two.[23] Therefore, in the specimens that exhibit high ductility like the 523 K-4-pass specimen, cross-slip from basal plane to non-basal plane as established by Koike *et al.*[24,25] might be in operation in addition to basal slip. On the other hand, in the specimens having texture (b) in which the basal plane is oriented parallel to the extrusion direction, the Schmid factors for basal slip is nearly 0 when the specimens are tensioned along the extrusion direction. Therefore, almost no shear force operates on the basal plane. As a result, it would be difficult for basal slip to occur. Consequently, the tensile strength and 0.2% proof stress would increase simultaneously with decrease of the elongation compared to the specimens having texture (a). This tendency is similar to the as-received specimen. Furthermore, in the 523 K-2-pass specimen that has mixed texture of (a) and (b), the 0.2% proof stress exhibits intermediate value between that of the specimens having textures (a) and (b).

Thus, the tensile strength and 0.2% proof stress of all specimens, as well as the elongation of the specimens having homogeneous microstructures, correspond well to their crystallographic orientations.

3.2 Changes in microstructures and tensile properties with annealing

The 8-pass as-ECAE specimens were annealed in order to investigate the changes in microstructures and mechanical properties with annealing.

3.2.1 Changes in hardness and microstructures

Figure 7 shows optical micrographs of the 8-pass as-ECAE and the annealed specimens, while the changes in Vickers hardness with annealing are shown in Fig. 8. Fine and equiaxed grains are homogeneously generated in the microstructure of the 8-pass as-ECAE specimen. This is as a result of the multiple pass ECAE processing at 473 K. The hardness decreases suddenly after annealing at 448 K for 1 h. The specimen annealed at 523 K for 1 h has fully softened, and the grains have coarsened up to average grain size of 4.3 µm with homogeneous microstructure as shown in Fig. 7. At this point, it is inferred that recrystallization driven by residual strain is complete. Therefore, the subsequent decrease of the hardness becomes gentle. Isochronal annealing for 4 h was carried out at higher temperatures in order to

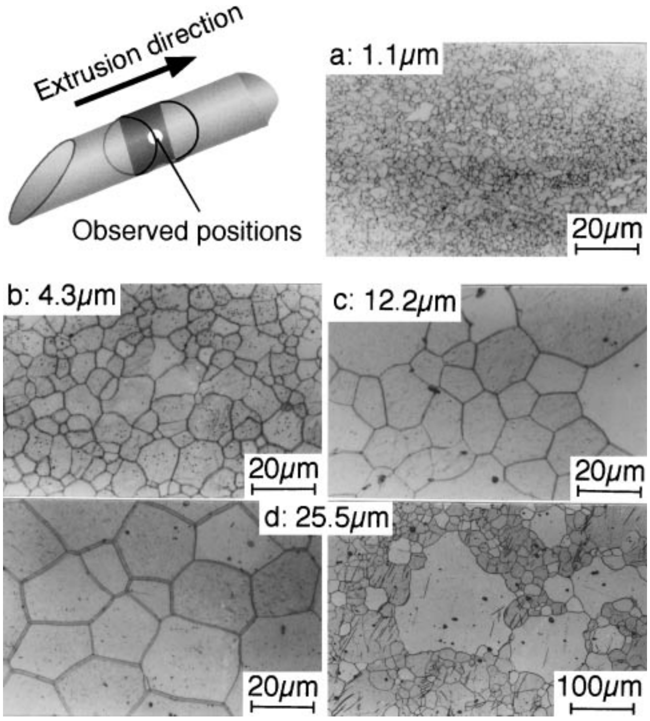

Fig. 7 Microstructures of (a) 8-pass ECAE-processed specimen and specimens subsequently annealed at (b) 523 K-1 h, (c) 723 K-4 h and (d) 773 K-4 h. Grain sizes are indicated in the figure.

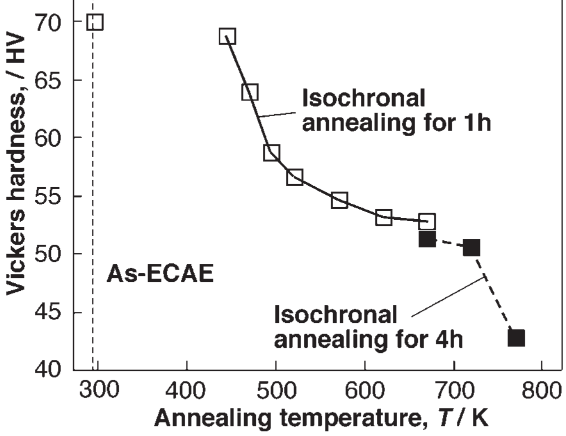

Fig. 8 Change in hardness with isochronal annealing.

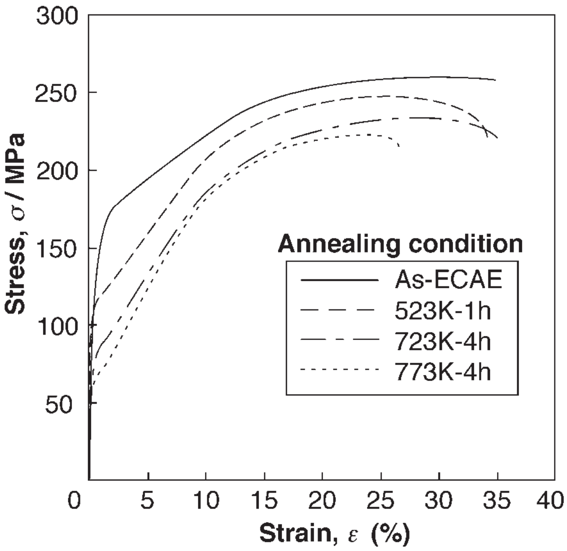

Fig. 9 Stress-strain curves of 8-pass as-ECAE and annealed specimens.

obtain further coarse grain size and microstructures. The microstructures and the changes in hardness are also shown in Figs. 7 and 8, respectively. The grain size of 723 K-4 h annealed specimen is coarser than that of the 523 K-1 h annealed specimen but the microstructure remains homogeneous. This result suggests that normal grain growth occurs up to relatively high temperatures in the investigated alloy. However, the grains of the 773 K-4 h annealed specimen coarsen up to average grain size of 25.5 μm with a large reduction of hardness as a result of abnormal grain growth.

3.2.2 Tensile properties

Figure 9 shows the stress-strain curves of 8-pass as-ECAE and annealed specimens. Both the tensile strength and 0.2% proof stress of all specimens decrease with increasing annealing temperature. That is, the strength of the specimens exhibits grain size dependencies. The specimens exhibit almost the same elongation, except the 773 K-4 h annealed specimen, which is lower. According to Koike *et al.*,[24] the cross-slip from basal to non-basal plane, as mentioned earlier, can be operative within the grain size range up to 10 μm. Therefore, in the 773 K-4 h annealed specimen having extraordinary coarse grains due to abnormal grain growth, the elongation would decrease.

3.2.3 Texture

Figure 10 shows the (0002) pole figures of the 8-pass as-ECAE and annealed specimens. Annealing reduces the intensities of preferred orientation. This tendency is similar to that of annealed AZ31 hot-rolled samples.[15] In the pole figure of 773 K-4 h annealed specimen, some high peaks, which may correspond to extraordinarily coarse grains, are observed inclined at about 10° to the extrusion direction. However, significant textural changes are not observed between the annealed specimens.

3.2.4 Grain size dependency

As expressed in section 3.1.2, the yield stress (σ_y) of polycrystalline metals usually depends on the grain size, according to the Hall-Petch relation as described below,

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

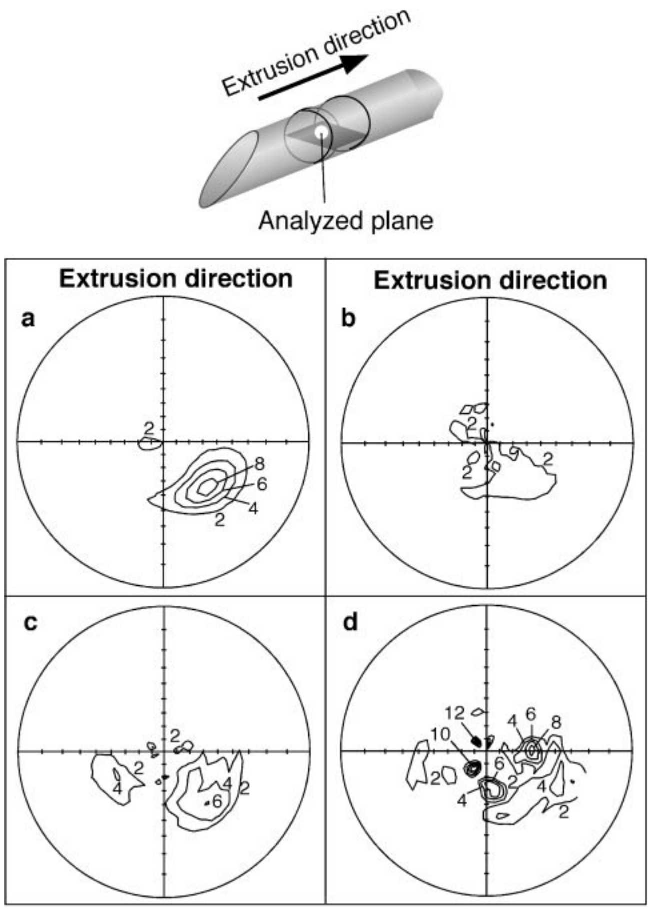

Fig. 10 (0002) pole figures of (a) 8-pass ECAE-processed specimen and specimens subsequently annealed at (b) 523 K-1 h, (c) 723 K-4 h and (d) 773 K-4 h.

where σ_0 is yield strength of single crystalline material, k is material constant, and d is grain size. Armstrong[5] proposed a "pile-up" model in which σ_0 and k of hcp materials relate to both CRSS for the easiest slip system and the CRSS for the slip system required to maintain the continuity of crystals at the grain boundaries when dislocations are accumulated at the grain boundaries. In addition, Rao *et al.*[7] supposed that σ_0 and k relate to CRSS for basal a slip and for prismatic a slip, respectively, and concluded that their orientation factors influence σ_0 and k. Thus, in magnesium alloys, the crystallographic orientation is extremely important during investigation of the grain size dependency of 0.2% proof stress. However, the grain size dependency dose not exist in the specimens having different textures as discussed in section 3.1.

Figure 11 shows the correlation between the grain size and the 0.2% proof stress of both the 8-pass as-ECAE and annealed specimens. In the annealed specimens having similar textures, the clear grain size dependency of 0.2% proof stress is exhibited according to Hall-Petch relation. Then σ_0 is 30 MPa and k is 0.17 MPa/m$^{-1/2}$. Koike *et al.*[26] have shown that σ_0 is approximately 60 MPa when the grain size is the only effective factor. That is, when the effects of precipitates and/or plastic strain are negligible. However, the σ_0 value obtained in the present study is half of the value proposed by Koike *et al.* It is expected that the basal **a** slip of

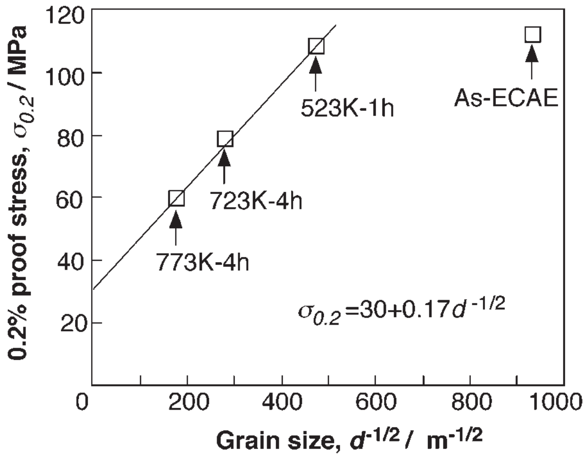

Fig. 11 Grain size dependencies of 0.2% proof stress of 8-pass ECAE-processed and annealed specimens.

the annealed specimens operates easily because most of the basal planes are inclined to the tensile axis. Thus, the easy basal slip brings about the reduction of the σ_0 value. On the other hand, the k value of 0.17 MPa/m$^{-1/2}$ corresponds to that obtained by Koike *et al.*[25)] Wilson[6)] and Rao *et al.*[7)] reported that the k value depends on crystallographic orientation and decreases when magnesium alloys have strong preferred orientation in their texture. Thus, further researches are required to clarify the correlation between texture and grain size dependencies of 0.2% proof stress.

The 0.2% proof stress of the 8-pass as-ECAE specimen is lower than the value expected from extrapolating the data for the annealed specimens. In this specimen, basal slip occurs easily because the intensity of the basal planes inclined to the tensile direction is higher than that of the annealed specimens. Therefore, cross-slip to non-basal plane may occur easily. This mechanism weakens the ability to obstruct dislocation motion. As a result, the grain size dependency might be reduced. Furthermore, Mabuchi *et al.*[12)] reported that the 0.2% proof stress of ECAE processed AZ91 alloy having average grain size of 0.5 µm is lower than the value obtained from Hall-Petch relation using other specimens. The specimen of the ECAE processed AZ91 alloy contains many low-angle grain boundaries due to low processing temperature of 448 K. The authors concluded that absorption of dislocations at the low-angle grain boundaries causes the reduction of the 0.2% proof stress. It is also deduced that the above mechanism reduces the 0.2% proof stress of the present 8-pass as-ECAE specimen because low-angle grain boundaries might be formed as a result of 8-pass ECAE processing at a relatively low temperature of 473 K.

As mentioned earlier, if the grain size dependency of tensile properties of magnesium alloys having strong crystal anisotropy is discussed, the effect of crystallographic orientation should be considered. In addition, other microstructural factors, for instance, dislocation density, amount of precipitates, grain boundary characteristics and so on, should also be considered. In the present study, the textures of the annealed specimens that exhibit clear grain size dependency are similar. Furthermore, it is inferred that the effect of dislocation density and the grain boundary characteristics on the 0.2% proof stress is small because the microstructures of the specimens are fully recrystallized. Consequently, it can be concluded the 0.2% proof stress of the annealed specimens depend essentially on grain size.

4. Conclusions

The correlation between ECAE processing conditions, microstructures including texture and tensile properties have been investigated in order to reveal the effect of the microstructural parameters on the strength and ductility of AZ31 magnesium alloy. The results are summarized as follows:

(1) The grain sizes of ECAE-processed specimens decrease with decreasing processing temperature. However, the microstructures are heterogeneous in the case of a small number of repetitive processing. A large number of repetitive processing at a low temperature is required to obtain fine and homogeneous grains.

(2) The tensile strength and 0.2% proof stress of as-ECAE processed specimens are largely influenced by crystallographic orientation rather than grain size. In 523 K-4-pass specimen, the 0.2% proof stress is reduced but elongation is significantly enhanced compared to those of as-received sample. This is because the specimen has a texture in which the basal plane is inclined at 45° to the tensile direction with a homogeneous microstructure. On the other hand, in the 4-pass specimen processed at 573 K, the 0.2% proof stress is higher and the elongation is lower because the basal plane is oriented parallel to the tensile direction.

(3) Intensified degrees of texture of 8-pass ECAE-processed specimens are weakened by annealing. The 0.2% proof stress of the annealed specimens decreases with increasing grain sizes, exhibiting clear grain size dependency according to Hall-Petch relation, that is, $\sigma_{0.2} = 30 + 0.17d^{-1/2}$.

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