Fabrication of fine recrystallized grains and their mechanical property in HPT processed pure magnesium

Mohit Joshi, Yuko Fukuta, Si Gao, Nokeun Park, Daisuke Terada, Nobuhiro Tsuji
Department of Materials Science and Engineering, Kyoto University, Japan
E-mail: mohit.joshi.87a@st.kyoyo-u.ac.jp

Abstract. To clarify the grain size effect on mechanical property in fine grained pure Mg, mechanical properties of fully recrystallized Mg with fine grain sizes were investigated. The specimens having fully recrystallized microstructures with mean grain sizes of 2.8 µm and 7.8 µm were fabricated by HPT processing and subsequent annealing. The 2.8 µm specimen showed discontinuous yielding with a high yield stress and low strain hardening while the 7.8 µm specimen showed continuous yielding with a low yield stress and high strain hardening. The as-HPT processed specimen showed a larger ductility than that in the subsequently annealed specimens.

1. Introduction
Magnesium and its alloys exhibit low formability and ductility at room temperature. The unfavorable mechanical properties are due to deficiency of independent easy slip systems because only basal slip is activated in Mg having HCP structure at room temperature [1]. Activation of other slip systems such as pyramidal slip system in Mg and Mg alloy can supply extra independent slip systems and can improve ductility.

In the previous research in our group on a commercial pure titanium (cp-Ti) with HCP structure, it was found that grain refinement down to approximately 1 µm improved yield strength without much decrease in uniform elongation. The behavior is different from that in cubic metals in which the increase in yield strength by grain refinement usually accompanies the sacrifice of ductility [2]. One possibility why the uniform elongation was maintained in fine-grained cp-Ti is the activation of various slip systems due to grain refinement. Since Mg belongs to HCP system, it is expected that grain refinement may lead to the improvement of mechanical properties due to activation of non-basal slips at room temperature.

Severe plastic deformation (SPD) processes, such as high pressure torsion (HPT), are one of the ways to obtain ultrafine grained microstructures with mean grain sizes much smaller than 1 µm. However, microstructures of SPD processed materials are simultaneously deformed structures including high-density dislocations. The dislocations affect the mechanical properties, and especially they are considered unfavorable for the ductility. Therefore, in order to clarify the grain size effects on the mechanical properties in pure Mg, fully recrystallized fine grains were fabricated by HPT processing and subsequent annealing and its mechanical properties were investigated in the present study.

2. Experimental Procedures
The material used for the present study was a pure magnesium with composition of 0.01%Al, <0.005%Cu, <0.005%Fe, 0.01%Mn, <0.005%Ni, 0.01%Si (purity of 99.9% in mass %). The as-received material was an extruded rod with a diameter of 25.4 mm.

Disks 10mm in diameter and 0.8mm in thickness were cut from the as-received material and then severely deformed by HPT process under a pressure of 6 GPa at a rotation speed of 0.2 rpm at room temperature. The samples were processed up to 1, 5, 10 or 15 rotations, which correspond to a shear strain of 19, 98, 196 or 294 respectively at the radial positions.
Corresponding to one half the radius of the disk, Vickers hardness of the HPT processed disks was measured. The top and bottom surfaces of the HPT processed disks for the hardness test were polished by buffing with diamond paste to obtain mirror-like finish. The hardness measurements were carried out with a load of 490mN (0.05 Kgf) and dwell time of 15 s. The hardness was measured at eight different positions 1 mm apart along radial directions from the disk center to the edge.

The disks HPT processed up to 5 rotations were annealed at 250 °C for 3 s or at 300 °C for 90 s in a oil bath furnace. Microstructures in the annealed disks were observed by electron back-scattering diffraction (EBSD) measurement. The EBSD samples were mechanically polished using ethanol and then chemically etched with a solution of 10% HNO₃, 20% HCl and 70% C₂H₅OH for 6 s.

Figure 1 (a) shows the position of the tensile specimens which were cut from the HPT processed and annealed disks at a radial distance of 2.5 mm from the disk center. Figure (b) shows the tensile direction of the specimens was perpendicular to the compressive direction of HPT process. The specimens had a gauge length of 2 mm, width of 0.9 mm and thickness of 0.45 mm. The surface of the tensile specimens was polished by buffing. In the tensile test, the specimens were set on a tensile testing machine with jigs, maintaining alignment of the testing machine and the small tensile specimens. A CCD camera and image analysis software were used to measure the displacement of the tensile samples because an extensometer was difficult to be attached due to small size of the specimens. The tensile tests were conducted at a strain rate of 1 x 10⁻³ s⁻¹ at room temperature.

3. Results and discussion

Figure 2 shows a grain boundary map of the starting material obtained from EBSD. Black lines indicate high angle grain boundaries with misorientation larger than 15°. The mean grain size calculated by linear intercept method was 33 µm.
Figure 3 shows the Vickers hardness along the radial direction of the specimens HPT processed up to 1, 5, 10 and 15 rotations, plotted as a function of the distance from the disk center. The hardness reduces with increase of the distance from the disk center. Hardness at the disk center showed the maximum value. As the number of rotations increases from 1 to 15, the hardness and its variation along radial distance reduce.

Figure 4 shows the hardness as a function of shear strain at all measurement points along the radial direction of the specimens. The shear strain by HPT process depends on the distance from the rotation axis (center) and thickness of the specimen. The shear strain (γ) can be calculated by the following equation.

$$\gamma = \frac{2\pi r N}{t}$$

where $r$ is the radial distance from the disk center, $t$ is thickness of the disk samples and $N$ is the number of rotation during the HPT process. In Fig. 3, it is found that the hardness reduces with increasing shear strain, and show a constant value of around 36HV above shear strain of approximately 80. The decrease in hardness value with increasing shear strain in pure magnesium has been reported [3], although it is an opposite tendency to the results reported in other kind of materials. In the report [3], softening with increasing shear strain was explained by an occurrence of recovery process during HPT.

Additionally, as the shear strain increases, the margin of error bars in hardness reduces. From the results, it is expected that the deformed microstructure above shear strain of 80 is homogeneous.
Shear strain of 80 corresponds to the strain at 2mm of distance from the center in the specimen HPT processed by 5 rotations. Therefore, the disks HPT processed up to 5 rotations were annealed in order to obtain fully recrystallized microstructures. Figure 5 shows grain boundary maps (a,c) at the distance of 2.5 mm from the center and inverse pole figures (b,d) showing orientations parallel to the normal direction (ND) of the disks HPT processed by 5 rotations and subsequently annealed at 250 °C for 3 s (a,b) and at 300 °C for 90 s (c,d). In Fig.3 (a), a fully recrystallized microstructure is observed and the mean grain size obtained by the linear intercept method was 2.8 µm. In Fig.3 (c), a fully recrystallized microstructure with a mean grain size of 7.8 µm was observed. In both specimens, it was confirmed that the mean grain size was nearly constant when the distance from the disk center was over 1.5 mm. The ND inverse pole figures (Fig.3(b) and Fig.2(d)) reveals that c-axis [0001] of most grains is parallel to ND which is the compression direction in the HPT process. Both samples have similar crystallographic textures and the texture of the annealed specimen is stronger. The results indicate that the specimens HPT processed and subsequently annealed show basal fiber texture.
The as-HPT specimen and the specimens HPT processed and subsequently annealed were used for the tensile test. Figures 6 shows the nominal stress – strain curves at room temperature with a strain rate of \(1 \times 10^{-3} \text{s}^{-1}\) for the fully recrystallized specimens with mean grain sizes of 2.8 µm, and 7.8 µm, and the specimen as-HPT processed up to 5 rotations. It is found that the strength of the as-HPT specimen is lower than that of the subsequently annealed specimens while the total elongation of the as-HPT specimen is larger than that of the annealed specimens.

The as-HPT specimen had a recovered structure [3] while the subsequently annealed specimens had fully recrystallized microstructures. Thus, it is expected that the dislocation density in the as-HPT specimen is higher than those in the subsequently annealed specimens. Therefore, the result that the strength of the annealed specimens was higher than that of the as-HPT specimen was surprising.

Among two annealed specimens, shapes of the stress-strain curves are quite different to each other. The specimen having the grain size of 2.8 µm specimen shows discontinuous yielding characterized by clear yield-drop, while the 7.8 µm specimen shows continuous yielding. Yield-point phenomena in ultrafine grained materials has been also reported in pure Al [2], pure Cu [4], pure Ti [5,6] and its alloys [4,7] and austenitic steels [8]. The yield stress of the 2.8 µm specimen (150 MPa) is larger than that of the 7.8 µm specimen (100 MPa). Furthermore, the 7.8 µm specimen exhibits large strain hardening after yielding, though the strain hardening of the 2.8 µm specimen is relatively low. The ultimate tensile strength of the 2.8 µm specimen (156 MPa) is lower than that of the 7.8 µm specimen (173 MPa).
MPa), due to difference in the strain hardening behavior. On the other hand, the total elongation of the specimens increases with decreasing the mean grain size. A summary of the mechanical properties is given in Table 1.

The effect of grain refinement on the strength and elongation seems peculiar. Comparison between the stress strain curves of the 2.8 mm specimen and as-HPT specimen suggests that free dislocations introduced by HPT processing play an important role for the mechanical properties. Similar phenomena have been reported in nanostructured metals as 'hardening by annealing and softening by deformation phenomena', which was explained by an effect of mobile dislocations in ultrafine grains [9]. For understanding these interesting results furthermore investigations in details are required.

![Nominal stress-strain curves of the specimens tensile-tested at room temperature and a strain rate of $1 \times 10^{-3}$ s$^{-1}$. The HPT processed and annealed specimens having fully recrystallized microstructures with average grain sizes of 2.8 µm & 7.8 µm, and the specimen HPT processed up to 5 rotations.](image)

**Fig. 6** Nominal stress-strain curves of the specimens tensile-tested at room temperature and a strain rate of $1 \times 10^{-3}$ s$^{-1}$. The HPT processed and annealed specimens having fully recrystallized microstructures with average grain sizes of 2.8 µm & 7.8 µm, and the specimen HPT processed up to 5 rotations.

**Table 1.** Summary of the mechanical properties of the present specimens obtained from the tensile curves.

| Specimens | Yield strength: $\sigma_y$ (MPa) | Tensile strength: $\sigma_{UTS}$ (MPa) | Uniform elongation: $e_u$ | Total elongation: $e_t$ |
|-----------|-------------------------------|---------------------------------|--------------------------|------------------------|
| 2.8 µm    | 150                           | 156                             | 0.20                      | 0.31                   |
| 7.8 µm    | 100 (0.2% proof stress)       | 173                             | 0.16                      | 0.21                   |
| 5 RHPT    | 114 (0.2% proof stress)       | 138                             | 0.07                      | 0.68                   |
5. Conclusions
Mechanical properties of a pure Mg severely deformed by HPT process and subsequently annealed were investigated. Major results obtained are summarized below.

1. The minimum mean grain size obtained as fully recrystallized microstructures was 2.8 µm in the present Mg specimens after the HPT process and subsequent annealing. A fully recrystallized specimen having a mean grain size of 7.8 µm was also fabricated.
2. The HPT processed specimens showed increase in the ultimate tensile strength and decrease in the total elongation by subsequent annealing.
3. The fully recrystallized specimen with a mean grain size of 2.8 µm showed discontinuous yielding characterized by a clear yield-drop and low strain hardening after yielding, while the 7.8 µm specimen showed continuous yielding and relatively high strain hardening. The elongation of the fully recrystallized specimens increased with decreasing the mean grain size.
4. The as deformed pure Mg specimen shows exceptionally high total ductility.

5. Acknowledgment
This research was financially supported by the Grant-in-Aid for Scientific Research on Innovative Area, “Bulk Nanostructured Metals” (area No. 2201), the Grant-in-Aid for Scientific Research (A) (No.24246114), and the Elements Strategy Initiative for Structural Materials (ESISM), through the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan (contact No. 22102002). The supports are gratefully appreciated. M.J. wishes to acknowledge the Japan International Cooperation Agency (JICA) for providing a doctoral scholarship under the JICA-FRIENDSHIP program.

References
[1] Tonda H and Ando S 2002 Metall. Mater. Trans. A 33 831
[2] Tsuji N, Ito Y, Saito Y and Minamino Y 2002 Scripta Mater. 47 893
[3] Edalati K, Yamamoto A, Horita Z and Ishihara T 2011 Scripta Mater. 64 880
[4] An X H, Wu S D, Zhang Z F, Figueiredo R B, Gao N and Langdon T G 2012 Scripta Mater. 66 227
[5] Terada D, Inoue M, Kitahara H and Tsuji N 2008 Mater. T 49 41
[6] Li Z, Fu L, Fu B and Shan A 2013 Mater. Lett. 96 1
[7] Sabirov I, Estrin Y, Barnett M R, Timokhina I and Hodgson P D 2008 Acta Mater. 56 2223
[8] Saha R, Ueji R and Tsuji N 2013 Scripta Mater. 68 813
[9] Huang X, Hansen N and Tsuji N 2006 Science 312 249