Anisotropic degradation behavior of moduli of extruded pure magnesium during low cyclic-tension fatigue

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Abstract. The mechanical properties of extruded pure magnesium during low cyclic-tension fatigue at room temperature were investigated in the extrusion and transverse loading directions (LDs) using ultrasonic reflection methods with longitudinal and shear waves (SWs), to clarify if there was an anisotropic degradation behavior between the extruded directions (EDs), which had not yet revealed. Regardless of the LD, the acoustic velocities and calculated Young’s and shear moduli decreased significantly with an increasing number of cycles because of the growth of voids at the grain and twin boundaries. An anisotropic behavior was revealed: when the deflection surface of the SWs was aligned in the LD, the amount of decrease in the moduli was greater than when the alignment was in the transverse direction (TD). Additionally, when the stress amplitude was adjusted to provide the same number of cycles to failure, the decrease in the moduli was somewhat greater when the LD was parallel to the extruded direction than to the TD. Longitudinal and SW propagation characteristics, and investigations of grain and twin boundaries after fatigue using electron backscatter diffraction (EBSD) based on field-emission scanning electron microscopy (FE-SEM), revealed that the most of the void-gap width was less than several nanometers (almost closed), which corresponded to the longitudinal wave amplitude. Other damaged-phase data were obtained using X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM) and Vickers-hardness test; the anisotropic degradation behaviors were attributed to the void morphology and the slight difference in orientation.

1. Introduction
Fatigue fracture often brings great social loss, and no evaluation method is sufficiently rigorous for field applications that could be applied to Stage I crack growth, in general which corresponds to most life of fatigue. Thus, a variety of approaches of nondestructive evaluation have been studied to detect slight changes of material during fatigue. For instance, eddy current [1], impedance-based monitoring [2], X-ray diffraction (XRD) [3], positron annihilation (PA) [4] and neutron diffraction (ND) [5] have been conducted. And for fretting fatigue, which occurs under a special condition of repeated relative surface motion and reduces the fatigue strength, the numerical simulations have been conducted [6-9].
On the other hand ultrasonic technique, which is relatively inexpensive and easy to use, have been also studied for long [10-12]. There are roughly two analyses methods in the technique, linear and non-linear. Linear method have been mostly applied for a concrete damage such as a crack. While, in recent years non-linear methods using harmonics have been becoming applied for fatigue evaluation due to its sensitiveness to dislocation motion [13-15]. There are also roughly two types of propagation modes in ultrasonic, longitudinal- and shear waves. In general, longitudinal wave is widely applied for both the linear- and non-linear methods due to its usability. Recently, the author of this article has applied shear wave to fatigue evaluation in nondestructive by use of linear- and non-linear methods, since shear wave is known to have highly sensitive interaction with a slight change of material, and indicated the characteristic behaviors accompanied with dislocation multiplication and residual stress during the fatigue [16-18].

Light metals, such as magnesium, are the most promising for structural applications in a low-carbon society. The strong texture of wrought magnesium makes it more difficult to form and the uncertainty in its fatigue lifetime limits its widespread use as a structural material.

Previous studies [19-21] used an ultrasonic pulse technique to non-destructively evaluate metal fatigue at room temperature. In that research, the elastic behavior of pure magnesium under low-, high- and giga-cycle tension fatigue was quantified when the loading direction (LD) was parallel to the extruded direction (ED). Significant reductions in the moduli were accompanied by fatigue damage. Most of the degradation was attributed to the presence of voids at the grain boundary. In this study, since the mechanical properties of expanded magnesium depend strongly on the ED, the anisotropy of the moduli behavior during low-cycle fatigue was evaluated by changing the LD : the validation was conducted using the already-known data of ED = LD, which was from Ref. [20], and newly acquired data of ED = TD. These results could contribute to a database allowing nondestructive inspection using this technique.

2. Experimental procedure

The material tested in this study was pure magnesium (Timminco Corp., Aurora, CO, USA). The chemical composition is shown in Table 1, and the mechanical properties of the material are listed in Table 2. The orientations obtained by X-ray diffraction (XRD) for the (0001) pole, using an incident slit width of 0.84 mm and a height of 3 mm, were 0.83 in the normal direction (ND), 0.07 in the ED and 0.09 in the transverse direction (TD). The configuration of the fatigue test sample is shown in Figure 1(a). The ED was oriented parallel to the LD (ED = LD specimen) and the TD (ED = TD specimen). The samples were subjected to cyclic tensile stress under controlled conditions. The stress ratio, R, was 0, and the frequency was 30 Hz, which was controlled by a hydraulic servo fatigue tester.
at room temperature. Figure 1(b) shows the cyclic stress as a function of the number of cycles to failure (S–N) for the material under the test conditions. The ultrasonic data of fatigue failure in the low cycles for ED = LD and ED = TD specimens were obtained under stress amplitudes of 28.4 MPa and 36.5 MPa, respectively. The specimens oriented as ED = TD were removed from the fatigue machine grip at arbitrary numbers of cycles to test the ultrasonic waveform. Under the ED = LD condition, the low-cycle samples’ stress amplitude, which was greater than the proof strength, made the specimen surfaces too rough to conduct ultrasonic measurements accurately. Hence, those fatigued specimens were separately prepared using four different numbers of cycles; the 69,478-cycle specimen was fatigue-fractured such that macroscopic cracks were evident in the evaluation area. Ultrasonic measurements were conducted before and after the fatigue tests; the ED = LD-fatigued specimens were polished again with 1,000-grit SiC paper for the measurements. The rates of change in the ultrasonic parameters were calculated by the fatigue progress of each sample. The ultrasonic data were collected until a normalized fatigue ratio, \( N/N_f \), of 1.0 was reached. The ultrasonic parameters of the as-extruded materials are listed in Table 3.

### Table 1. Chemical composition of the magnesium material (mass%).

| Material   | Al  | Zn  | Mn  | Fe  | Ni  | Cu  | Si  | Pb  | Ca  | Sn  | Cd  | Mg  |
|------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Pure Mg    | 0.004 | 0.006 | 0.0074 | 0.0021 | 0.0007 | 0.004 | 0.003 | 0.001 | 0.001 | <0.001 | <0.0001 | Bal. |

### Table 2. Mechanical properties of the material.

| Temper          | 0.2% Proof stress (MPa) | Tensile strength (MPa) | Elongation (%) |
|-----------------|-------------------------|------------------------|---------------|
| as-extruded (ED = LD) | 53                       | 147                     | 9.6           |
| as-extruded (ED = TD) | 131                      | 187                     | 2.8           |

### Table 3. Acoustic properties of the as-extruded material.

| Deflected surface of SVs | Longitudinal wave velocity \((V_l/m\cdot s^{-1})\) | Shear wave velocity \((V_s/m\cdot s^{-1})\) | Young’s modulus \((E/GPa)\) | Shear modulus \((G/GPa)\) | Poisson’s ratio \((\nu)\) |
|-------------------------|-----------------------------|--------------------------|-----------------|-----------------|-----------------|
| ED                      | 5585                        | 3132                     | 43.38            | 17.07           | 0.271           |
| TD                      | 5585                        | 3081                     | 42.33            | 16.52           | 0.281           |

To evaluate variation in the elastic moduli as fatigue progressed, the longitudinal and shear wave (SW) velocities in the middle of the samples (Figure 1(a)) were measured separately using longitudinal wave and vertically polarized SW transducers, with a center frequency set to 5 MHz. The
diameters of the longitudinal and SW probes were 4 and 10 mm, respectively. The transducers made contact with the sample under a load of 55 N using water-free naphthenic hydrocarbon oil at room temperature. The deflected surface of the SWs was aligned in the LD and TD. Ultrasonic wave pattern analysis was performed using an elastic modulus system (UMS-H; Toshiba Tungaloy, Kawasaki, Japan). The input voltage, sampling frequency and averaging number were 100 V, 100 MHz, and 16, respectively. The measuring accuracy of the system is within 0.1 % in velocity.

The detailed procedure is described in Ref. [19]. The wave velocities, i.e., the longitudinal \( V_l \) and shear \( V_s \), gave the Young’s modulus, \( E \), and the shear modulus, \( G \), according to the following relationships [22], where \( \rho \) is the density of the sample:

\[
E = \frac{3 \rho V_s^2 \left( V_l^2 - \frac{4}{3} V_s^2 \right)}{V_l^2 - V_s^2}
\]

\[
G = \rho V_s^2
\]

The structural variations in the material during the fatigue process were evaluated using the ultrasonic evaluation samples. After fatigue failure, the fracture surface and cross-sectional texture in the LD were examined using scanning electron microscopy (SEM), optical microscopy (OM) and an electron backscatteringdiffraction pattern (EBSD) method based on field-emission scanning electron microscopy (FE-SEM). In addition, using the specimens with different number of cycles the Vickers hardness were measured at the center of the samples in the LD. The numbers for averaging were 10 points.

Figure 1. (a) Configuration of the fatigue test sample (dimensions in mm). The thickness of the sample was 5.5 mm. (b) S–N curve of the material \((R = 0)\).
3. Results

The change ratios for the velocities of the longitudinal waves and SWs, as a function of $N/N_f$ during the low-cycle fatigue, are shown in Figure 2(a) and (b), respectively. The thick and slim arrows in the graphs indicate the ED and the directions of the deflected surface of the SWs, respectively. The results reveal the following two tendencies that did not depend on the ED. First, for a given specimen, the SW velocity decreased to a greater extent than the longitudinal wave. Second, for the same specimen, the SW velocity decreased more when the deflected surface was aligned with the LD than with the TD. Furthermore, although the cycle to fracture was almost same, by comparing the two behaviors of the $ED = LD$ and $ED = TD$ specimens, the following tendency, which depended on the ED, was revealed. The velocities of the longitudinal and SWs decreased more when the LD was parallel to the ED ($ED = LD$ specimen) than in the transverse setting ($ED = TD$ specimen). Notably, the stress amplitude of the $ED = LD$ specimen was 22% less than that of the $ED = TD$ specimen.

Figure 3(a) and (b) shows the change ratios for the Young’s and shear moduli, respectively, resulting from the velocities as a function of the $N/N_f$ during the low-cycle fatigue. The thick and slim arrows in the graphs indicate the ED and the directions of the deflected surfaces of the SWs, respectively. As well as the velocities, the results reveal the following two tendencies that did not depend on the ED. First, for a given specimen, the shear modulus decreased slightly more than the Young’s modulus. Second, for the same specimen, the Young’s and shear moduli decreased more when the deflected surface of the SWs was aligned in the LD rather than in the TD. Furthermore, by comparing the two behaviors of the $ED = LD$ and $ED = TD$ cases, the following tendency, which depended on the ED, was revealed. When the LD was parallel to the ED ($ED = LD$ specimen), the Young’s and shear moduli decreased more than with the transverse setting ($ED = TD$ specimen).

![Figure 2](image_url). Change ratios during low-cycle fatigue for the velocities of the (a) longitudinal waves and (b) shear waves (SWs) as a function of the normalized fatigue ratio, $N/N_f$ (DS: deflected surface).
Figure 3. Change ratios during low-cycle fatigue for the (a) Young’s modulus and (b) shear modulus as a function of $N/N_f$ (DS: deflected surface).

The fracture surfaces near the crack initiation site of both loading modes, ED = LD and ED = TD, are shown in Figure 4. All of the surfaces exhibited crack propagation at the grain boundary, i.e., the grain boundary was considerably degraded during both fatigue modes. Optical micrographs of the cross-sections of the LD of the ED = LD specimen, and the EBSD analysis of the LD of the ED = TD specimen, are shown in Figure 5(a) and (b), respectively. Both materials showed interface voids at the grain or twin boundaries that corresponded to the LD, and which were mainly normal to the principal stress or oriented at 45° to it. However, recognizable voids with openings in the micrometer-range were few in total in a 10-mm-wide view. Figure 6 shows the variation of the Vickers hardness with the increasing number of fatigue cycles. The ED = LD specimen indicated a work-hardening behavior in the early cycles of fatigue. On the other hand the ED = TD specimen relatively stayed unchanged during the early period.

Figure 4. Scanning electron microscopy (SEM) images of the fracture surface near the crack initiation site.
Figure 5. (a) Optical microscopy (OM) image and (b) electron backscatter diffraction (EBSD) analysis of the cross-sections in the loading direction (LD) for the extruded direction (ED) and transverse direction (TD) specimens, respectively (GB = grain boundary, TB = twin boundary).

Figure 6. Variations of the Vickers hardness of the (a) ED = LD specimen and (b) ED = TD specimen.
4. Discussion

4.1. Anisotropy derived from defect morphology (anisotropy did not depend on the ED)

First, we consider the cause of the large decrease in the velocities with fatigue damage (Figure 2). This mechanical degradation, i.e., the several percentage-point decrease in velocities and resulting moduli, cannot be explained by common lattice defects, such as strain, twin, or dislocations; the influence of the velocity was estimated at less than 1 % [20]. Thus, voids at the grain and twin boundaries evident in Figure 5 were considered as the cause of the degradation. However, since the recognized voids were quite few, the defect that grew under the fatigue modes was thought to be mainly in a closed state [21]. In fact, regarding the grain-boundary damage, the deformation mechanism map of pure magnesium [23] indicates the possibility of grain-boundary creep under the fatigue test condition, even at room temperature. Furthermore, the two velocity anisotropies noted in Figure 2 support the boundary degradation concept resulting from the growth of a void. First, as shown in Figure 7(a), a longitudinal wave propagates a nanometer-sized gap in a state of compressive stress. In principle, a SW cannot propagate such a void, even if it is completely closed because of a lack of elastic restoration. Thus, under the development of voids, the defect is influenced largely by the SWs rather than by the longitudinal waves. Second, under uniaxial loading, a diffusional void can be dynamically induced in the normal plane against the LD. Thus, as shown in Figure 7(b), the SW propagates more easily, with a deflected surface aligned in the TD rather than in the LD (Figure 2(b)).

The behaviors mentioned above are anisotropies derived from the defect morphology, which depended on the LD and not on the ED; this was recognized for both ED = LD and ED = TD specimens. The anisotropic behavior of the moduli calculated from the velocities was the same. Concerning the slight difference between the Young’s and shear moduli, it is clear from Equations (1) and (2) that the shear modulus is more affected by an SW.

![Diagram](image)

**Figure 7.** Propagation advantage between (a) longitudinal and SWs and (b) SWs for which the deflected surfaces are aligned in the LD and TD.
4.2. Anisotropy derived from texture (anisotropy depended on the ED)

The anisotropies discussed in the previous section derived from the void morphology that depended on the LD, not on the ED. On the other hand, the observed anisotropic behavior, whereby the velocities and moduli decreased more when the LD was parallel to the ED (ED = LD specimen) than in the transverse setting (ED = TD specimen), cannot be explained by the defect morphology. This anisotropy is considered to result from the texture, which was determined by the ED.

The development mechanism of a void during the fatigue test is now considered. In pure magnesium, for which the grain-boundary diffusion coefficient is relatively high [24], Coble creep, grain-boundary sliding (GBS) or cleavage crack originating from the two creeps could contribute to the interface degradation. However, there is a conflict, i.e., although the development of all of these boundary defects is dependent on the driving stress, the decrement of the mechanical properties was much larger in the ED = LD specimen, for which the stress amplitude was 22 % lower, than in the ED = TD specimen. The following is a plausible explanation. The value of the critical resolved shear stress (CRSS) of magnesium suggests that a basal slip is the dominant slip system at room temperature [25]. The specimen in this fatigue test had a strong texture; the c-axis was oriented in the through-thickness direction, yet the orientation was slanted slightly to the ED (ND: 0.83, ED: 0.09, TD: 0.07). Since the strong texture restricts the slip system considerably, the slight difference of c-axis orientation between the ED and TD led to the different mechanical behaviors. Considering the effect of shear stress on basal slip in this test, it was noted that the Schmidt factor of the ED = LD specimen was higher than that of the ED = TD specimen (Figure 8). The data of Table 2 suggest that the anisotropy of the mechanical properties could result from the orientation difference. In fact, as shown in Figure 6 the difference in behavior of the variations of Vickers-hardness supports this: the data of ED = LD specimen show explicit work-hardening due to a basal slip. Facile basal slip in the ED = LD specimen would result in the accumulation of dislocations at the grain or twin boundaries, leading to stress concentrations at the boundaries. Thus, a relatively small driving stress would lead to significant degradation at the interfaces. Nevertheless, the cause of the anisotropy that depended on the ED remains incompletely understood; with verification of quantitative repeatability the relationship between the morphology of lattice defects and fatigue progress, and an analysis of the Schmidt factor of the texture, are required.
5. Conclusions

The mechanical properties of extruded pure magnesium during low cyclic-tension fatigue were investigated at room temperature, in the extruded and transverse LDs, using ultrasonic reflection methods with longitudinal waves and SWs. The following anisotropies were found:

For a given specimen,

- Both longitudinal and SW velocities decreased significantly with fatigue progress. Comparing the two wave modes, the decrease was clearly greater for the SW than for the longitudinal wave. The resulting Young’s and shear moduli, calculated from the velocities, also decreased significantly. The decrease in the shear modulus was slightly greater than that of the Young’s modulus, because the shear modulus is more strongly affected by SWs.
- The SW velocity decreased more when the deflected surface was aligned with the LD than with the TD. The resulting moduli were similar.

These anisotropies were attributed to the defect morphology.

At nearly the same number of cycles to failure, the ED = LD and ED = TD orientations showed the following anisotropies:
When the LD was parallel to the ED (ED = LD specimen), although the applied stress was 22% smaller, the decrease in the velocities and resulting moduli was greater than in the transverse setting (ED = TD specimen).

This anisotropy was attributed to the slight orientation of the texture.

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