Effect of Grain Size on Fatigue Behavior in AZ61 Mg Alloys Fabricated by MDFing

Yoshihiko Uematsu1,2, Toshifumi Kakiuchi1, Hiromi Miura2 and Taishi Nozaki3

1Department of Mechanical Engineering, Gifu University, Gifu 501–1193, Japan
2Department of Mechanical Engineering, Toyohashi University of Technology, Toyohashi 441–8580, Japan
3Kawasaki Heavy Industries, Ltd., Tokyo 105–8315, Japan

Magnesium (Mg) alloy AZ61 was multi-directionally forged (MDFed) under decreasing temperature conditions using a die. The average grain size decreased with increasing MDFing pass number. The initial grains size of 21.6 μm in the as-annealed specimen decreased gradually during MDFing and an average grain size of 0.3 μm could be attained after MDFing for 8 passes. The tensile strength and Vickers hardness were improved with increasing pass number from 1 to 8. Hall-Petch relationship was held for those static mechanical properties. Subsequently, tension-tension axial loading fatigue tests were performed using the as-annealed specimen and MDFed ones to 1, 3, 6 and 8 passes in which cumulative strains were 0.8, 2.4, 4.8 and 6.4 respectively. Fatigue strengths were highly improved by MDFing with increasing pass number of forging from 1 to 3. However, the improvement looked almost saturated over the pass number of 3. The observed breaking-up of the Hall-Petch relationship concerning with fatigue limits was attributed to grain-boundary sliding followed by crack initiation and propagation along grain boundaries.

1. Introduction

Magnesium (Mg) alloys possess the highest specific strengths among the structural metallic materials, and are beneficial to the weight saving of transportation systems. The weight saving of transportation systems is very important for high fuel efficiency. Consequently, it is expected to replace the structural components made of aluminum (Al) alloys or steels by Mg alloys. However, hardness, 0.2% proof stress and tensile strengths of Mg alloys are still lower than conventional high-strength Al alloys such as 7000 series Al alloys. It is well known that grain refinement is effective to improve mechanical properties of materials. Recently, many severe plastic deformation (SPD) methods are proposed to achieve mechanical properties of materials. Materials are forged with changing forging direction during multi-directional forging (MDFing) technique, materials are forged with changing forging direction for 90 degrees pass by pass. Other SPD methods, such as equal channel angular pressing (ECAP) and high pressure torsion (HPT) are also applied to Mg alloys to achieve ultrafine-grained (UFGed) structures. However, rather large components could be fabricated only by MDF, and that is one of the advantages of MDFing. To use UFGed materials for mechanical components, understanding of their fatigue behavior is very important indeed. For example, Vinogradov et al. revealed that the fatigue limit of ZK60 Mg alloy could be enhanced by ECAP three times higher than the as-received material. However, detailed analyses of fatigue fracture behavior of UFGed Mg alloys are still insufficient, and the fatigue properties of MDFed Mg alloys have not been reported yet.

In the present study, MDFing was applied to AZ61 Mg alloy to obtain bulky UFGed specimens. Tension-tension axial fatigue tests were conducted on MDFed AZ61 Mg alloys. Various specimens having different grain sizes from 21.6 μm down to 0.3 μm were prepared by changing the number of MDFing passes and the effect of grain size on the fatigue behavior was systematically investigated.

2. Experimental Procedure

2.1 Material and MDFing procedure

The material used is AZ61 Mg alloy, whose chemical compositions (mass. %) are as follows; Al: 5.9, Zn: 0.66, Mn: 0.3, Cu: 0.002, Si: 0.009, Fe: 0.002, Ni: 0.002, Mg: balance. Rectangular-shaped samples with dimensions of 15 mm × 22.2 mm × 33.3 mm were cut from as-hot-extruded bar, followed by annealing at 733 K for 1 h to have average grain size of 21.6 μm. The annealed samples were MDFed using a die equipped with a heater at an initial strain rate of 3.0 × 10−3 s−1. Pass strains Δε = 0.8 were employed. Therefore, applied cumulative strains after 1, 2, · · · , 8 passes of MDFing were 0.8, 1.6, · · · , 6.4. The MDFing temperature was gradually decreased pass by pass of forging from 603 K down to 393 K. The details of MDFing procedure of the used specimens in the present study were described elsewhere.

In the present study, samples MDFed to 1, 3, 6 and 8 passes in addition to as-annealed one were prepared for evaluation. The microstructure and basic mechanical properties of MDFed AZ61Mg alloy have been reported by Miura et al. They performed tensile tests under different strain rates and temperatures. The grain sizes and tensile strengths at ε = 1.0 × 10−1 s−1 and at room temperature of the MDFed AZ61Mg alloys are summarized in Table 1. The same material was used in this study. Vickers hardness of the specimens was originally re-measured by the present authors using micro-Vickers hardness tester at a load of 2.942 N with holding time of 15 s. The results are also revealed in Table 1. The re-measured hardness was nearly the same with that obtained by Miura et al. Changes in hardness and grain sizes as a function of cumulative strain are shown in Fig. 1. The grain size decreases and hardness increases with increasing cumulative strain, namely number of pass. Miura et al. have reported linear relationship between hardness and grain size d−1/2.
of the MDFed AZ61Mg alloy\(^7\)). They also reported that same tendency was observed between yield stress and grain size \(d^{-1/2}\) excepting as-annealed specimen which had higher yield stress than 3-passes MDFed specimen due to texture strengthening effect\(^7\)). Therefore, Hall-Petch relationships were held concerning with static mechanical properties.

### 2.2 Fatigue test procedures

Fatigue specimen configuration is shown in Fig. 2. The specimen has the gauge section with the length of 2 mm, width of 3 mm and thickness of 2 mm. As schematically shown in Fig. 3, five pieces of specimens were cut out from one MDFed block, where longitudinal direction was perpendicular to the final forging axis. Fatigue specimens were polished by emery paper and mirror finished by buffing before fatigue tests. Axial loading fatigue tests were performed using 49 kN electro-hydraulic fatigue testing machine. Load wave form was sinusoidal with the frequency \(f = 20\) Hz and stress ratio \(R = 0.1\). In this case, tension-tension loading cycles \((R = 0.1)\) were applied to avoid buckling of the specimens with the thickness of 2 mm (Fig. 2).

### 3. Results and Discussion

#### 3.1 Fatigue test results

\(S-N\) curves of the specimens as-annealed, MDFed to 1, 3, 6 and 8 passes are revealed in Fig. 4. Fatigue strengths were highly improved in the specimens MDFed to 1 and 3 passes compared with the as-annealed one, and fatigue strength increased with increasing pass number. In the specimens MDFed to 6 and 8 passes, however, fatigue strengths were comparable to or slightly lower than those MDFed to 3 passes. The fatigue limit, \(\sigma_w\), is defined as the run-out stress level at \(10^7\) stress cycles. Accordingly, \(\sigma_w\) was 45, 70, 85, 80, 78 MPa for the specimens as-annealed, MDFed to 1, 3, 6 and 8 passes, respectively.

Figures 5–7 reveals typical fracture surfaces and magnified views near crack initiation sites in the specimens as-annealed, MDFed to 3 and 8 passes, respectively. All the specimens show flat fracture surfaces, indicating that fatigue cracks basically initiated due to cyclic slip deformation. Defect-domi-
nated fatigue crack initiation was not recognized. It should be noted that the specimen MDFed to 8 passes composed of UFGs (d = 0.3 μm) exhibits much flatter fracture surface near the crack initiation site than the as-annealed one as shown in the magnified views of Fig. 5(b) and Fig. 7(b).

In the MDFed specimens, Hall-Petch relation is held in tensile strength and hardness\(^7\) as shown in Fig. 8. Vinogradov et al. revealed that Hall-Petch relation was found in fatigue limits, \(\sigma_w\), of ECAPed commercially pure Ti\(^15\). The fatigue limits of the MDFed AZ61Mg alloys are shown in Fig. 9 as a function of \(d^{1/2}\). In the specimens as-annealed, MDFed to 1 and 3 passes, fatigue limit abruptly increases with decreasing grain size. The increase in not linear, but Hall-Petch-like relation is still held. However, in the specimens MDFed to 6 and 8 passes, the fatigue limits are nearly comparable to that of the specimen MDFed to 3 passes. The samples as-annealed, MDFed to 3, 6 and 8 passes hold Hall-Petch relation in view of hardness and tensile strength (static mechanical properties) as already shown in Fig. 8. It would indicate that stress cycling have detrimental effect on the fatigue strengths of the specimens MDFed to 6 and 8 passes having UFGed structures. Consequently, some expected influential factors on fatigue strengths will be discussed in the following sections.

### 3.2 Texture

It is well known that fatigue behavior is sensitive to the microstructure of specimens. And strong texture could be formed in Mg alloys during plastic deformation because the activated slip systems at ambient temperature are limited to basal slip. Thus XRD analyses by using SmartLab (RIGAKU) were performed on the final forging plane to evaluate texture evolution of basal plane. Figure 10 shows the pole figures of basal plane (0002) in each specimen, where TD direction corresponds to the longitudinal direction of the specimen. Due to the annealing process, the texture is not so strong in the as-annealed specimen, but the basal plane slightly oriented to RD direction. However, in the specimens MDFed to 1 and 3 passes, basal planes are weakly oriented to RD direction.

The texture intensities of basal plane are shown in terms of MDFing pass number in Fig. 11(a). Basically, the intensity decreased with increasing pass number. Yang et al.\(^5\) have revealed that the crystal orientation of grains of MDFed AZ31 Mg alloy was
more randomized with increasing pass number of cold MDFing with pass strains of 0.1. Thus it is reasonable that the texture was weakened with increasing MDFing pass number as shown in Fig. 11(a). The strong texture in the as-annealed specimen induced texture-strengthening effect to raise the yield strength. Consequently, the crystal orientation randomization of microstructure in the specimens MDFed to 6 and 8 passes could result in the lower fatigue strengths. Nevertheless, it should be emphasized that the specimen MDFed to 3 passes exhibits weaker texture, but still higher fatigue limit than the as-annealed one. Furthermore, Fig. 11(b) summarizes the relationship between fatigue limits and MDFing pass number. The change of fatigue limit in terms of MDFing pass number, i.e., grain size, described in Fig. 11(b) does not simply correspond to the change in terms of texture intensity in Fig. 11(a). It is evident, therefore, that the large change in texture intensity (Fig. 11(a)) is not directly correlated with the change in the fatigue limit (Fig. 11(b)). Thus it could be concluded that the texture change in the specimens MDFed to 6 and 8 passes were not closely related to the breaking-up of Hall-Petch relationship shown in Fig. 9.

3.3 Grain growth during fatigue loading

If the microstructures in MDFed materials are unstable, grain growth due to dynamic recrystallization could be possible during fatigue loading, which could trigger the invalidity of Hall-Petch relationship. Thus similar to the texture evaluation, XRD analyses were performed on the final forging plane to obtain rocking curves (RCs). When the microstructure contains large grains, relatively clear peak would appear in RCs. Figure 12 reveals RCs of each specimen. In the specimens as-annealed and MDFed to 1 pass, clear peaks are recognized in the curves, while the curves become flatter with increasing MDFing pass number. It should be noted that the vertical axis of Fig. 12(e) is magnified compared to Figs. 12(a)–12(d). Figure 13 represents the difference between maximum and minimum peaks of RCs in the specimens MDFed to 3, 6 and
8 passes. The difference decreases with increasing pass number. Those results qualitatively indicate that the peak in the rocking curve decreased with decreasing grain size.

Subsequently, RCs were obtained using fatigue specimens before and after fatigue tests. Using the specimens MDFed to 6 and 8 passes, RCs were obtained at the gage section in the initial condition without fatigue loading. Subsequently, fatigue tests were conducted at the fatigue-limit stress levels of each sample, namely 80 and 78 MPa for the specimens MDFed to 6 and 8 passes, respectively. Both fatigue tests were terminated at 10^7 stress cycles, and then RCs were measured again using specimens with fatigue damage. Figure 14 shows RCs before and after fatigue tests. It should be noted that any peaks did not newly appear after fatigue loading. Therefore, it could be concluded that grain growth to the size of 1.5 μm (the average grain size of the specimen MDFed to 3 passes) did not occur during fatigue loading up to 10^7 cycles in the specimens MDFed to 6 and 8 passes.

3.4 Grain-boundary sliding (GBS)

As revealed in the previous report\(^7\), static stress-strain diagram of MDFed specimens composed of UFGs showed dependency on strain rate even at room temperature. Actually, the yield stresses of the specimens MDFed to 6 and 8 passes decreased with decreasing strain rate from 1 × 10^{-4} s^{-1} to 1 × 10^{-4} s^{-1}, which could be attributed to grain-boundary sliding (GBS)\(^7\). It means that GBS could occur even at room temperature at low strain rate region around 1 × 10^{-4} s^{-1}. In the fatigue tests, load frequency was 20 Hz and it was too fast for GBS to occur. However, strain wave form is sinussoidal, and consequently the period, in which the strain rate slower than 1 × 10^{-4} s^{-1}, could appear in the strain wave form as schematically drawn in Fig. 15. The strain wave form could be transferred from the stress wave form because all load-controlled fatigue tests were conducted under elastic limits. Thus the time, when the strain rate was slower than 1 × 10^{-4} s^{-1}, could be integrated to 10^7 stress cycles, namely to the fatigue limit. The total time for 10^7 cycles was estimated to be around 2 min. The time for tensile test at 1 × 10^{-4} s^{-1} to rupture was around 20 min. The total time, when the strain rate was slower than 1 × 10^{-4} s^{-1} during fatigue tests, was shorter than the tensile test (20 min). However, GBS possibly takes place even at higher strain rate and at shorter period of time\(^7\). Occurrence of GBS could induce stress concentration especially at triple junctions and grain-boundary facets to accelerate crack initiation. That is, the finer grain size becomes, the easier GBS becomes to induce stress concentration and preferential crack initiation. GBS, therefore, could be one of the affecting factors on fatigue strengths even at 10^7 cycles.

3.5 Environmental effect

All fatigue tests were conducted in air-conditioned laboratory air. It is known that the fatigue strengths or fatigue crack propagation rates could be affected by the humidity because properties of Mg alloys are sensitive to environment atmosphere. Especially, Uematsu et al. revealed that hydrogen diffusion into Mg alloy could accelerate fatigue crack growth rates\(^17,18\). There is a possibility that the sensitivity against hydrogen was drastically enhanced in UFGed structures because grain boundary could be dominant hydrogen diffusion pass. Therefore, an environmental chamber was integrated into the fatigue testing machine, and some fatigue tests were conducted with supplying dry air. In this case, the dew point of supplied dry air was 213 K (−60°C). In Fig. 16, the fatigue strengths in dry air of the specimens MDFed to 6 passes are shown. It should be noted that the fatigue strengths in dry air (solid symbols) are higher than those in laboratory air (open
The lower fatigue strengths in laboratory air could be attributed to the effect of corrosion on the specimen surface and subsequent diffusion of hydrogen into the material\(^{18}\). Quantitatively, fatigue limits are 80 MPa and 105 MPa in laboratory air and in dry air, respectively. The fatigue limit was improved 25 MPa (namely 31\%) in dry air by eliminating environmental effect. It means that if fatigue test was conducted in dry air using the as-annealed specimen, the improvement of fatigue limit by eliminating environment effect should be less than the specimen MDFed to 6 passes. Because the hypothesis is that environmental sensitivity is lower in the as-annealed specimen than in the specimen MDFed to 6 passes with UFGs. Based on this hypothesis, a fatigue test was conducted in dry air using the as-annealed specimen. The stress level was set to be 70 MPa, so that 25 MPa (56\%) higher than the fatigue limit in laboratory air with humidity. If the hypothesis is true, the specimen should fail, in other words, the improvement of fatigue limit by eliminating environment effect would be less than 25 MPa. However, fatigue test at 70 MPa ran out at \(10^7\) cycles as shown in Fig. 16. It indicates that the sensitivity against environment of the MDFed specimen to 6 passes was not higher than the as-annealed one, revealing that environment effect was not responsible for the breaking-up of Hall-Petch relationship in the UFGed specimens MDFed to 6 and 8 passes.

3.6 Grain size irregularity in distribution

Fatigue crack initiation is strongly dominated by grain size. The average grain size of MDFed specimens to high cumulative strain regions was small, as 0.5 \(\mu m\) after 6 passes and 0.3 \(\mu m\) after 8 passes of MDFing. But if the grain size distribution is wide in the MDFed specimens, and the microstructure contains a few large grains, fatigue crack could preferentially initiate in those irregularly large grains. Therefore, the grain size distributions of the specimens as-annealed and MDFed to 3, 6 and 8 passes were statistically measured by line-intercept method, and histograms of grain size distribution are shown in Fig. 17. It should be noted that the specimen MDFed to 3 passes could contain some grains larger than 3 \(\mu m\). But the largest grain sizes in the specimens MDFed to 6 and 8 passes are still smaller than 1.6 \(\mu m\) and 1.2 \(\mu m\), respectively. Thus grain size irregularity in distribution could not be related to the comparable fatigue strengths of the specimens MDFed to 6 and 8 passes to that MDFed to 3 passes.

3.7 Specimen surface morphology before and after fatigue test

Finally, the assumption about GBS is considered more in detail. To find the trace of occurrence of GBS, the specimen surface morphology before and after fatigue test was precisely investigated. Figure 18 reveals specimen surfaces of the as-annealed specimen before (Fig. 18(a)) and after (Fig. 18(b)) fatigue test (\(\sigma_a = 45\) MPa, run-out specimen). Before fatigue test, the specimen surface has some parallel lines evolved during surface polishing procedure. Even after the application of cyclic stress, specimen surface morphology is similar to that before fatigue test. On the other hand, Fig. 19 shows clear change before and after fatigue test (\(\sigma_a = 85\) MPa, \(N_f = 2.9 \times 10^6\)) in the surface morphology on the specimen MDFed to 6 passes. Before fatigue test (Fig. 19(a)), specimen surface was smooth, and the parallel lines due to polishing procedure were less clear than on the as-annealed specimen (Fig. 18(a)). Figures 19(b) and 19(c) reveal that the surface near fatigue fracture surface was roughened by stress cycling. It should be noted that the surface roughness shown in Fig. 19(c) was nearly comparable to the average grain size of the specimen MDFed to 6 passes (\(d = 0.5\) \(\mu m\)). This fact implies occurrence of GBS during cyclic loading. Thus it was found that the specimen morphology after stress cycling was dependent on the grain size (Figs. 18(b), 19(b) and 19(c)). Because GBS becomes easier with decreasing grain size, stress concentration evolved at triple junction and grain-boundary facets should be more emphasized to induce earlier crack initiation. Furthermore, Fig. 20 reveals typical
fatigue fracture surfaces near crack initiation site of the specimens MDFed to 1, 3 and 6 passes. Flat areas are recognized in the specimens MDFed to 1 (Fig. 20(a)) and 3 (Fig. 20(b)). However, the fracture surface of the specimen MDFed to 8 passes has fine roughness where the roughness was almost comparable to the grain sizes, implying grain boundary crack growth.

Based on the discussion described in the sections 3.2~3.6, it could be concluded that texture, grain growth, environment effect and grain size irregularity were not decisively responsible for the breaking-up of Hall-Petch relationship in fatigue limit. However, Figs. 18 and 19 revealed the different surface morphology between the specimens as-annealed and MDFed to 6 passes under fatigue loading. As already mentioned in the section 3.4, GBS could be possible to occur during fatigue loading. It is considered that GBS in the specimen MDFed to 6 passes occurred and resulted in the roughened surface, in which quantitative roughness was nearly comparable to the average grain size (Fig. 19(c)). In addition, the specimen MDFed to 8 passes exhibited fatigue fracture surface with fine roughness (Fig. 20(c)). Consequently, the surface roughness induced by GBS and following fatigue crack initiation along grain boundary might be the main factor for the breaking-up of Hall-Petch relationship in terms of fatigue limit.

Actually, it is reported that crack propagation along grain boundary in the UFGed AZ61Mg largely spoiled impact toughness.19)

Such breaking-up of Hall-Petch relationship in static mechanical properties is sometimes reported and the critical grain size is estimated to be around 100 nm.20) The average grain sizes in the AZ61 Mg alloys MDFed to high cumulative strain regions (i.e., MDFed to 6 and 8 passes) were 0.5 and 0.3 μm, which are rather coarser than the above indicated condition. It was reported by Koike et al.,21,22) that non-basal slips could be activated with decreasing grain size. Therefore, there still exits the possibility of non-basal slip activation in the specimens MDFed to 6 and 8 passes. Thus it is assumed that the breaking-up phenomenon of Hall-Petch relationship at coarser grain size in UFGed Mg alloys might be related to the complicated interactions of various effects of texture, grain growth, GBS, environment, grain size irregularity, non-basal slip, crack propagation along grain boundary and etc., even if the effect of GBS is a major controlling factor.

4. Conclusions

Multi-directional forging (MDFing) was applied to AZ61 Mg alloy and various specimens having different grain sizes were prepared by MDFing to 1, 3, 6 and 8 passes. The samples were fatigue tested under conditions of tension-tension axial loading. The effects of grain size on the fatigue behavior were investigated. Based on the experimental results, the following conclusions could be yielded:

(1) Notable grain refinement took place during MDFing. Grain size decreased gradually with increasing MDFing pass number and reached 0.3 μm after 8 passes of MDFing, i.e., to cumulative strain of 6.4. Hall-Petch relationship was held in the static mechanical properties such as hardness and tensile strength in the MDFed specimens.

(2) Fatigue strengths were improved by MDFing. Fatigue strengths increased dramatically with increasing MDFing pass number from 1 to 3. However, further MDFing to 6 and 8 passes could not enhance the fatigue strengths. Hall-Petch relationship was, therefore, not held in the fatigue limits of the specimens MDFed to 6 and 8 passes to have average grain sizes of 0.5 and 0.3 μm, respectively.

(3) XRD analyses, fatigue tests in dry air condition and grain size distribution estimation had revealed that texture, grain growth, environment effect and grain size irregularity were not decisively responsible for the breaking-up of Hall-Petch relationship concerning with fatigue limit.

(4) The strain rate estimation and surface roughness analyses before and after fatigue loading suggested the activation of grain-boundary sliding (GBS) in the samples MDFed to 6 and 8 passes to have ultrafine-grained structures. It was considered that the breaking-up of Hall-Petch relationship in fatigue limit could be related to the activation of GBS followed by fatigue crack initiation and growth along grain boundary.

Acknowledgement

This work was financially supported by JSPS Grant-in-Aids for Scientific Research on Innovative Area “Bulk Nanostructured Metals” (Area No. 2201).
REFERENCES


Effect of Grain Size on Fatigue Behavior in AZ61 Mg Alloys Fabricated by MDFing 1461