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Unraveling cyclic deformation mechanisms of a rolled magnesium alloy using in situ neutron diffraction

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Abstract—In the current study, the deformation mechanisms of a rolled magnesium alloy were investigated under cyclic loading using real-time in situ neutron diffraction under a continuous-loading condition. The relationship between the macroscopic cyclic deformation behavior and the microscopic response at the grain level was established. The neutron diffraction results indicate that more and more grains are involved in the twinning and detwinning deformation process with the increase of fatigue cycles. The residual twins appear in the early fatigue life, which is responsible for the cyclic hardening behavior. The asymmetric shape of the hysteresis loop is attributed to the early exhaustion of the detwinning process during compression, which leads to the activation of dislocation slips and rapid strain-hardening. The critical resolved shear stress for the activation of tensile twinning closely depends on the residual strain developed during cyclic loading. In the cycle before the sample fractured, the dislocation slips became active in tension, although the sample was not fully twinned. The increased dislocation density leads to the rise of the stress concentration at weak spots, which is believed to be the main reason for the fatigue failure. The deformation history greatly influences the deformation mechanisms of hexagonal-close-packed-structured magnesium alloy during cyclic loading.
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Keywords: Magnesium alloy; Cyclic loading; Deformation mechanisms; Neutron diffraction; Twinning

1. Introduction

Magnesium (Mg) alloys have attracted much attention from the automobile and aircraft industries over the last two decades due to their low density, high specific strength, excellent thermal conductivity and extraordinary damping capacity [1–4]. Mg alloys are the lightest metal commercially available that can be used as structural materials. For applications such as load-bearing components, it is vital to understand the fatigue behavior of Mg alloys when considering reliability and durability. In cast Mg alloys, defects, such as casting porosity and inclusions, which commonly act as crack sources, are harmful to fatigue properties, and may assist fatigue-crack initiation, reduce lifetime and decrease cyclic strength [5,6]. On the contrary, wrought Mg alloys are basically defect-free, and thus exhibit better fatigue properties than casting alloys [7–9]. Moreover, the study of the cyclic behavior of wrought Mg alloys may shed light on the intrinsic fatigue mechanisms of Mg alloys [10]. Therefore, mechanistic understanding of the cyclic-deformation behavior can facilitate the practical applications of wrought Mg alloys as structural materials.

Five independent slip systems are necessary in order to achieve arbitrary homogenous deformation for polycrystalline materials. Mg has a hexagonal-close-packed (hcp) structure with a limited number of slip systems. In principle, Mg has five independent slip systems, including two (00.2) $\langle 11.0 \rangle$ slip (or basal $\langle a \rangle$ slip), one (10.0) $\langle 11.0 \rangle$ slip (or prismatic $\langle a \rangle$ slip), one (10.1) $\langle 11.0 \rangle$ slip (or pyramidal $\langle a \rangle$ slip) and one (11.2) $\langle 11.3 \rangle$ slip (or pyramidal $\langle c + a \rangle$ slip) [11–13]. The pyramidal $\langle c + a \rangle$ slip is the only one that can provide strain along the c-axis. However, it is very difficult to activate the pyramidal $\langle c + a \rangle$ slip at room temperature, due to the high CRSS [14]. Thus, deformation twinning, along with slip systems, contributes to satisfying the von Mises yield criterion, but it can only offer a limited amount of strain. It has been recognized that the tension–compression asymmetry of wrought Mg alloys results from the unidirectional nature of the deformation twinning [12,15].

Because the c/a ratio of the hcp-structured Mg is 1.624, which is less than the ideal hard-sphere value, $\sqrt{3}$, the most prominent twinning system is {10.2} $\langle 10.1 \rangle$ tensile twinning (or extension twinning), which provides a tensile strain along the c-axis [12,13,15,16]. The {10.2} $\langle 10.1 \rangle$ tensile twinning leads to a sudden reorientation of the matrix lattice ∼86.3° by a tensile stress along the c-axis or a compressive stress perpendicular to the c-axis [17,18]. After tensile

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twinning, the detwinning process could be easily activated by a stress reverse. The alternate twinning and detwinning process has often been reported in previous studies during strain-path changes and/or cyclic loading in the highly textured wrought Mg alloys [17–22].

Comprehensive studies have concentrated on the effects of strain amplitude [23–25], mean stress [26–28], strain ratio [8,9,28,29], strain rate [29], microstructure [8,30,31], grain size [32,33], rare earth elements [34,35], hysteresis energy [26], heat-treatment [36], temperature [5], environment [37,38] and initial texture [39–42] on the fully reversed strain-controlled low-cycle fatigue behavior of the wrought Mg alloys. Moreover, in the last decade, progress has been made in theoretical modeling to predict the slip, twinning, and detwinning behavior in the hcp-structured material during strain-path changes and cyclic loading [11,22,39,43–47]. Overall, the low-cycle fatigue life of the wrought Mg alloys increases with the decrease of strain amplitude, strain ratio and mean stress, as well as the increase of strain rate. The previous fatigue studies [7,9,23,39,40,48] demonstrated that the strain–life curves can be described well by the Basquin and Manson–Coffin equations, as well as the Holloman relation. Recently, it has been found that a clear kink point around a total strain of 0.005 can be observed from the strain amplitude– fatigue life curve in the extruded AZ61 and ZK60 Mg alloys, indicating that the dislocation and twinning–detwinning deformation modes were dominant below and above the 0.005 total strain, respectively [28,30,49]. Subsequently, the twinning and detwinning processes were usually involved in the fully reversed strain-controlled low-cycle fatigue at a relatively large total strain (> 0.005), which has been proven experimentally by optical microscopy [8], in situ electron backscatter diffraction (EBSD) [50], in situ transmission electron microscopy (TEM) [21,51] and in situ neutron diffraction measurements [17,18,39,52,53]. In general, the alternate twinning and detwinning process during cyclic loading is thought to be the main reason for the strong tension–compression asymmetry of the hysteresis loops. However, in the prior in situ experiments, discontinuous step-loading methods were commonly employed, which contain only a limited number of measurements. Usually, a few data points (at peak strain, zero stress and zero strain) per fatigue cycle were presented in the previous research [17,18,39,52]. Certainly, these limited data points are representative on the hysteresis loop in each fatigue cycle, but the information missing between two measurements is inevitable. For example, in one cycle how the twinning/detwinning behavior evolves and when the transition of twinning/detwinning to dislocation deformation occurs, etc., are not clearly represented by those scarce measurements, and most importantly how those behaviors evolve during fatigue cycling before the material fails are not answered clearly by the previous studies.

In this investigation, the above questions will be addressed by state-of-the-art real-time in situ neutron diffraction. Previously, we have reported the effects of strain-path changes and pre-deformation history on the deformation dynamics and mechanisms of a rolled AZ31B Mg alloy investigated by real-time in situ neutron diffraction under a continuous loading condition [20,54]. In the present research, the influence of cyclic loading on plastic deformation, and twinning and detwinning behavior, including the residual-twin evolution during the fully reversed low-cycle fatigue, are presented here in-depth by employing the same experimental method.

2. Experimental

2.1. Experimental materials

A commercial rolled AZ31B Mg alloy plate (chemical composition: 3 wt.% Al, 1 wt.% Zn and Mg as balance) in H24 temper (strain-hardened and partially annealed) was selected for the current research; it contains a typical rolling texture with the crystal *c*-axis parallel to the normal direction (ND) and perpendicular to the rolling direction (RD) of the rolling plate [55]. The thickness of the rolled plate is 76 mm, which facilitates the machining of low-cycle fatigue samples along the through-thickness direction (or ND). Dog-bone cylindrical low-cycle fatigue specimens with an 8 mm diameter and 16 mm gage length were fabricated, according to the American Society for Testing and Materials (ASTM) Standard E606-04. After sample machining, all the specimens were annealed at 345 °C for 2 h to relieve the existing residual stress. The average grain size of the annealed samples is ~40 μm.

2.2. Low-cycle fatigue experiment

The fully reversed strain-controlled low-cycle fatigue experiment was conducted at a total strain amplitude, ±0.02, at room temperature with triangular loading waveforms. The fatigue cycles, 1st, 2nd, 5th, 10th, 20th, 50th and 70th cycles, were selected for the real-time in situ neutron diffraction measurements before the sample failed in the 71st fatigue cycle. The strain rate in the selected cycles was relatively low for the real-time in situ neutron diffraction measurements. Specifically, the strain rate in the 1st cycle was $6.8 \times 10^{-6}\ \mathrm{s}^{-1}$, in the 2nd cycle was $5.4 \times 10^{-6}\ \mathrm{s}^{-1}$ and in the other chosen cycles was $8.3 \times 10^{-6}\ \mathrm{s}^{-1}$. Except for the fatigue cycles chosen for the neutron diffraction measurements, the frequency was 1 Hz with a corresponding strain rate of $8 \times 10^{-2}\ \mathrm{s}^{-1}$.

2.3. Real-time in situ neutron diffraction measurements

The in situ neutron diffraction measurements were performed at the VULCAN Engineering Materials Diffractometer [56–58], the Spallation Neutron Source (SNS), Oak Ridge National Laboratory (ORNL) during fully reversed low-cycle fatigue under a continuous-loading condition. Due to the high neutron flux at the VULCAN instrument at SNS, the real-time in situ neutron diffraction measurements have been successfully employed in a number of studies [20,54,59–62]. The experimental setup for the Mg alloys neutron diffraction measurements under mechanical loading is described in detail elsewhere [20,54]. In the following, a brief description is provided. The sample was mounted horizontally to the VULCAN load frame, with the axial direction parallel to the ND and the horizontal-radial direction along RD. The angle between the incoming neutron beam and the specimen was 45°. The two stationary detector banks are located at ± 90° to the incoming beam. In this setup, two complete diffraction patterns (the axial (ND) and horizontal-radial (RD) directions with diffraction vectors parallel ($Q\|$) and

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