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Cyclic deformation and anelastic behavior of ZEK100 magnesium alloy: Effect of strain ratio

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ABSTRACT

Wrought magnesium alloys with low rare-earth (RE) contents are being considered for lightweight automotive applications. The purpose of this study was to identify the effect of strain ratio on cyclic deformation behavior of a rolled ZEK100-O Mg alloy with 0.2 wt% neodymium. The microstructure in its annealing condition consisted of equiaxed grains which were oriented with most *c*-axes perpendicular to the rolling direction. This alloy exhibited a superior combination of tensile strength and ductility due to its weak basal texture and decreased stacking fault energy. Significant plastic deformation occurred in the tensile phase of the first cycle at strain ratios of $R_\varepsilon = 0$ and 0.5. The asymmetry of initial hysteresis loops tended to diminish towards the mid-life cycles. With increasing strain ratio, fatigue life first increased, reached its maximum at a strain ratio of $R_\varepsilon = -1$, and then decreased. This was attributed to a combined effect of mid-life stress amplitude, plastic strain amplitude and mean stress, where the stress amplitude increased and plastic strain amplitude decreased with increasing strain ratio. The closer the strain ratio to $R_\varepsilon = -1$ was, the lower the absolute value of mean stress was. The mean stress relaxation occurred mainly in the initial stage and for the strain ratios more remotely from $R_\varepsilon = -1$. The anelastic behavior of this alloy largely remained arising from the twinning and detwinning, with the strain ratio identified as an influential parameter via sensitivity analyses. The anelastic strain amplitude, along with three newly-defined parameters (eccentricity, angle deviation, and relative slope change) all decreased with increasing strain ratio, reflecting more symmetric hysteresis loops.

1. Introduction

One key approach in improving fuel efficiency and reducing climate-changing CO_2 emissions is vehicle lightweighting [1–6]. A great deal of research curiosity has been attracted by the alternatives towards decreasing the fuel consumption of passenger vehicles, known to be among the most abundant means of transportation [7]. Lightweight Mg alloys, characterized by their low density, high strength-to-weight ratio and superior damping capacity, are of special interest in the transportation industry. Meanwhile, numerous limitations are still encountered in the structural applications of Mg alloys (e.g., high directional anisotropy and poor formability at room temperature). Tension–compression yield asymmetry and mechanical anisotropy in wrought magnesium alloys can be related to the development of strong texture in the deformation process [8–10]. In fact, mechanical twinning plays a considerable role in the deformation of Mg alloys due to its hexagonal close-packed (hcp) crystal structure with limited modes of deformation [11–13]. It is possible to improve the tension–compression yield asymmetry and the room temperature formability by the addition of alloying elements especially rare-earth (RE) elements. This has the advantage of introducing texture randomization during rolling and extrusion processes [14–16]. In addition, safe design and structural applications of Mg alloys are in need of cyclic deformation and fatigue behavior due to the dynamic loading that is experienced in service [13,17–21].

The fatigue resistance of rare-earth containing Mg alloys has been reported in some studies in the literature. Fu et al. [22] focused on the low cycle fatigue (LCF) behavior of AZ91D Mg alloy while changing the amount of Ce. Wang et al. [23] considered an extruded GW83 (Mg–8.0Gd–3.0Y–0.5Zr) alloy by studying the reversed strain-controlled tension–compression loading along the extrusion direction. Yang et al. [25] performed very high cycle fatigue tests of Mg–12Gd–3Y–0.5Zr alloy, and introduced a relieved tension–compression yield asymmetry in addition to improved fatigue failure resistance. The above mentioned RE-rich magnesium alloys are relatively expensive due to a large amount of RE elements added; nevertheless, the cost of materials is an important consideration in the automotive sector since affordability for the general public needs to be taken into account.

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The effect of strain ratio on cyclic deformation and fatigue has also been a subject of some investigations. Yu et al. [26] conducted strain-controlled fatigue experiments on an extruded AZ61A Mg alloy at three strain ratios ($R_\varepsilon = -\infty$, -1 and 0). A strong cyclic hardening characteristic was observed with decreasing strain ratio. Patel et al. [27] evaluated strain-controlled cyclic deformation behavior and fatigue life of a super-vacuum die cast AM60B alloy at two strain ratios ($R_\varepsilon = -1$ and 0.1). A longer fatigue life was reported at the lower strain ratio. In the same context, Lin et al. [28] studied the effect of strain ratio on the fatigue behavior of hot-rolled AZ91 Mg alloy and linked the enhanced fatigue life to the low strain ratio. Begum et al. [19] identified the influences of both strain ratio and strain rate on cyclic deformation and fatigue life of an AZ31 extruded Mg alloy. They reported more asymmetric hysteresis loops as the strain ratio decreased due to the increasing compressive strain applied.

The current study involves a comparatively low Nd-containing (i.e., 0.2 wt% Nd) ZEK100 rolled magnesium sheet alloy that was recently developed. Min et al. [29] studied the formability of this alloy in a two-stage forming process with intermediate annealing. Niu et al. [30] reported that ZEK100 alloy possessed superior warm formability over AZ31B alloy, indicating the potential for the volume production of magnesium automotive parts. This alloy also exhibited superior true fracture strains (i.e., up to 60% higher than the commercial AZ31B alloy for all strain paths at all temperatures and strain rates applied [31]). It was reported that the basal texture was weakened due to the Nd addition in ZEK100 alloy, however the anisotropy in the tensile properties still remained [32]. While some limited microstructural studies on ZEK100 were reported in the literature [29,33], EBSD (electron backscatter diffraction) investigations along the three different observational planes of the ZEK100 sheet, are not available to the authors' knowledge. No studies about the effect of strain ratio on the cyclic deformation characteristics of ZEK100 alloy are available so far. It is unclear how the hysteresis loops in the initial and mid-life cycles evolve while changing strain ratios, and to what extent the tension–compression yield asymmetry would remain. Information on the pseudo-elastic or anelastic behavior of this alloy is also lacking, including how it could be properly quantified and what the main influencing parameters are. Therefore, the main objective of this study was to evaluate cyclic deformation and pseudo-elastic behavior in relation to the strain ratio in the low RE-containing ZEK100 alloy.

2. Material and experimental procedure

The investigated material in the current study was a recently-developed ZEK100 magnesium alloy in the forms of rolled sheets (3 mm in thickness), with a composition (in wt%) of 1.3Zn, 0.25Zr, 0.2Nd, 0.01Mn and Mg (balance) [33], which was supplied by Magnesium Elektron via the University of Waterloo and Magna International Inc. The alloy was in the annealed condition designated as ZEK100-O (500 °C for 15 min in an electrical oven [29,31]). An optical microscope (OM) equipped with an image analysis system, and a scanning electron microscope (SEM) JSM-6380LV along with an Oxford energy dispersive X-ray spectroscopy (EDS) system, were used to observe microstructures. EBSD measurements were carried out at a step size of 0.8 μm on three different observational planes of the as-rolled alloy and a step size of 0.6 μm in the areas near fracture surfaces by means of Oxford integrated AZtecHKL advanced EBSD system with NordlysMax^2 and AZtecSynergy along with a large area analytical silicon drift detector. For microstructural observations, sample preparations were accomplished by following standard metallographic techniques, and microstructural features were revealed using an etchant of acetic picral solution (i.e., 4.2 g picric acid, 10 ml acetic acid, 10 ml H_2O and 70 ml ethanol). Specimens for the EBSD examinations were first mechanically polished and then electro-polished in an electrolyte of 10 ml nitric acid and 40 ml ethanol for about 35 s at 20 V and room temperature. Crystallographic texture was determined with a PANalytical X-ray diffractometer (XRD) with Cu K_α radiation at 45 kV and 40 mA in a back reflection mode by measuring partial pole figures (i.e., ranging between $\Psi = 0°$ and 75°). Texture data were subsequently evaluated using the Matlab-based MTEX software [34]. The correction of the defocusing coming from the rotation of the XRD sample holder was made using the experimental data from magnesium powder diffraction. EBSD data analyses were conducted by means of the AZtecHKL EBSD data acquisition software by Oxford Instruments.

Tensile testing was carried out using a computerized United tensile machine with a sample gauge length of 25 mm (or a parallel length of 32 mm) at a strain rate of $1 \times 10^{-2}\ s^{-1}$. Strain-controlled "pull-push" low cycle fatigue (LCF) tests were conducted using a computerized Instron 8801 fatigue testing system at room temperature via LCF3 program at a fixed strain rate of $1 \times 10^{-2}\ s^{-1}$ with a triangular loading waveform. The fatigue samples (Fig. 1(a)) were machined with the loading axis parallel to the rolling direction (RD). The dependence of the LCF characteristics of ZEK100 alloy on the strain ratio was studied at a given strain amplitude of 0.8%. This strain amplitude was selected based on the previously conducted LCF experiments at a strain ratio of $R_\varepsilon = -1$ [35] where at smaller strain amplitudes (e.g., 0.2–0.4%), very thin and hardly detectable stress–strain hysteresis loops, approaching nearly elastic deformation were detected, while at higher strain amplitudes (e.g., 1.0%, 1.2%), wide hysteresis loops were obtained but with a very short fatigue life. In this context, different values of $R_\varepsilon = 0.5$, 0, -1, -3, and $-\infty$ were chosen so as to have relatively uniformly-distributed hysteresis loops along the strain axis. The strain ratio selection was also justified by the sensitivity analysis of the anelastic behavior that will be presented in the coming sections, and for which a wider range of strain ratios would be more significant. A minimum of two samples were tested at each strain ratio in order to make sure of the reproducibility of the results. Fracture surfaces of fatigued samples were examined by means of SEM, to identify different features such as fatigue initiation sites and propagation mechanisms. Special attention was also given to the eventual appearance of residual twins in the neighboring regions to the fracture surface of the fatigued samples by means of optical microscopy and EBSD evaluations.

3. Results

3.1. Microstructure and EBSD measurements

Samples cut along three observational planes of the ZEK100 alloy sheet were separately prepared for the purpose of EBSD investigations. A three-dimensional EBSD band contrast (BC) mapping is shown in Fig. 1(b), where RD, TD and ND stand for the rolling, transverse, and normal directions, respectively. It should be noted that the BC mapping results in Fig. 1(b) were evaluated independently, without grain averaging or other type of interpolations. Black dots pointed in Fig. 1(b) indicate the presence of a small fraction of randomly distributed second-phase precipitates. Our previously-conducted XRD phase identification and energy dispersive X-ray spectroscopy (EDS) analysis revealed that the particles contained Mg, Zn and Nd in the form of MgZn and $Mg_{12}Nd$ intermetallic compounds [35]. These results were also consistent with other studies on the same or similar alloys [14,36,37].