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Achieving excellent superplasticity in an ultrafine-grained QE22 alloy at both high strain rate and low-temperature regimes

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ABSTRACT

The high-temperature tensile deformation behavior of ultrafine-grained (UFG) QE22 alloy developed by friction stir processing was investigated at various strain rates ($5 \times 10^{-4} - 1 \times 10^{-2}$ s^{-1}) and temperatures (300–450 °C). The UFG QE22 alloy exhibited excellent high strain rate superplasticity with the maximum elongation of 1630% at a temperature of 450 °C and strain rate of 1×10^{-2} s^{-1}, which is the highest elongation reported till date in QE22 alloy. The UFG QE22 alloy also shows superior low-temperature superplasticity with a maximum elongation of 850% at a temperature of 350 °C and strain rate of 3×10^{-3} s^{-1}. Thus, the developed UFG microstructure is found to demonstrate dual-mode superplasticity; both at high strain rate and low-temperature. The primary UFG microstructure and basal texture developed via multi-pass FSP, along with pinning action of $Mg_{12}Nd$ eutectic and fine nano precipitates of $Mg_{12}Nd_2Ag$ at grain boundaries played a major role in contributing to the dual-mode superplasticity. The cavitation behavior of the UFG QE22 alloy during superplastic deformation in a dual-mode regime has been studied. The kinetics of superplastic deformation of UFG QE22 alloy was estimated and found to be significantly faster as compared to conventional magnesium alloys processed by various severe plastic deformation routes.

1. Introduction

Magnesium (Mg) alloys are potential materials for automotive and aerospace applications due to their attractive properties viz., lightweight, high strength-to-weight ratio and high damping resistance [1]. However, the hexagonal lattice of Mg alloys limits their usage due to low formability at room temperature [2,3]. At high temperatures, additional slip systems are activated in the Mg alloys and promote superplasticity which is an attractive option for forming Mg alloys [4].

Superplasticity refers to the ability of a crystalline material to exhibit large strains (Elongation >200%) when subjected to high-temperature tensile deformation. This phenomenon is not only a topic of academic interest but also of industrial significance due to its capability of forming intricate parts from materials that are hard-to-form [5]. Development of superplasticity in Mg alloys has drawn a considerable attention over the last decade. The fine-grained microstructure is a fundamental requirement to exhibit superplastic behavior in the Mg alloys [6]. Among Mg alloys, the Mg-Al-Zn alloys are widely used in industries. In general Mg-Al-Zn alloys shows better superplasticity at low-temperatures (≤350 °C) and low strain rates (≤10^{-3} s^{-1}) resulting in the increase of processing time to complete the superplastic forming operations. Therefore, the commercial feasibility of superplasticity in Mg-Al-Zn alloys is limited.

The applicability of Mg alloys in superplastic forming can be enhanced by developing a suitable microstructure, which can sustain high strain rate superplasticity (HSRS). HSRS is the superplasticity occurring at strain rates at or above 10^{-3} s^{-1} [5,7]. To realize HSRS in Mg alloys, the microstructure should have fine grains, preferably ultrafine-grained (UFG) or nanostructured materials. Several reports discussed the challenges of achieving HSRS in conventional Mg alloys processed by various severe plastic deformation routes (equal channel angular pressing/extrusion (ECAP/E), high-ratio differential speed rolling (HRDSR), high-pressure torsion (HPT), hot extrusion (EXT), indirect extrusion and hot rolling (HR) process) despite of attaining UFG size [8–25]. This is due to the lack of thermal stability of UFG Mg alloys, which is very critical to superplastic temperatures [12,26,27]. Generally, HSRS is observed at relatively high-temperatures of ~0.8 T_m, where

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T_m is the melting point of the material [5]. Prolonged exposure of UFG Mg alloys at high-temperatures makes their microstructure thermally unstable resulting in anomalous or abnormal grain growth. Thus, a specific microstructure that holds high thermal stability while attaining HSRS is essential to overcome the above-mentioned limitations. In order to achieve high thermal stability in UFG Mg alloys, rare earth (RE) elements like Gd, Nd, Y etc., are often added to the Mg matrix [26–31]. Addition of RE elements to Mg matrix can be beneficial in two ways: (i) thermal stability of UFG Mg alloys increases, which result in enhancement of superplasticity and (ii) the formability during high-temperature tensile deformation improves by weakening its texture formed during severe plastic deformation process [32–37].

Hence, the desirable microstructure of Mg alloys should possess the following important characteristics to achieve HSRS (i) stable UFG microstructure under the given conditions of temperature, time and stress for deformation [38,39]. (ii) equiaxed with well-defined equilibrium high-angle boundaries. Both requirements can be satisfied by choosing an appropriate combination of Mg-RE alloy and the type of severe plastic deformation technique.

Many researchers have studied the superplastic behavior of Mg-RE alloys processed by various severe plastic deformation techniques [26,27,40–51]. Recently, T.Y Kwak et al. [46] have studied the superplastic behavior of the HRDSR processed Mg-13Zn-1.5Y alloy and reported maximum elongation of 1021% at low-temperature and strain rate of 1×10^{-3} s^{-1}. Similarly, W.J Kim et al. [52] have also reported the low-temperature superplasticity (888%) in a HRDSR processed Mg-9.25Zn-1.66Y alloy at a strain rate 1×10^{-3} s^{-1}. The observed superplasticity behavior was attributed to the fine dispersion of second phase particles, which suppressed the grain growth by effective pinning on grain boundaries. The superplastic characteristics of a Mg-Gd-Y-Zr alloy processed by multi-directional impact forging have been investigated at various strain rates and temperatures [48]. The maximum elongation of 300% was obtained at a temperature of 450 °C and strain rate of 5×10^{-4} s^{-1}. G. Cao et al. have observed the HSRS in a submerged friction stir processed Mg-Y-Nd alloy with an elongation of 549% at a temperature of 485 °C and strain rate of 1×10^{-1} s^{-1} [50].

Among various Mg-RE alloys, QE22 alloy is the most famous because of its very good thermal stability [53], remarkable age hardening response, excellent creep resistance, decent tensile and fatigue strength [30]. The high-temperature deformation behavior of QE22 alloy processed by different thermo-mechanical treatments (hot extrusion followed by age hardening treatments and other thermal treatments) is extensively studied by the group led by Ryspaev [41,44,54–56]. However, a thorough analysis on the governing mechanisms and kinetics of superplastic behavior (both in HSRS and low-temperature superplasticity (LTS) domain) of friction stir processed (FSP) QE22 alloy, especially in UFG regime is still unavailable. Therefore, the present work is aimed to: (i) study the superplastic behavior of UFG QE22 alloy both in HSRS and LTS regime and establish its governing mechanisms; (ii) correlate the superplastic deformation behavior with detailed microstructural and texture information to understand the cavitation behavior of UFG QE22 alloy; and (iii) establish the superplastic deformation kinetics of UFG QE22 alloy. Finally, the superplastic data of the UFG QE22 alloy is compared with the conventional Mg alloys and Mg-RE alloys processed using various severe plastic deformation routes.

2. Experimental procedures

2.1. Materials and processing

Rare earth (Nd) contained Mg alloy (QE22) provided by Magnesium Elektron, with a composition of Ag, 2.08 wt%; Nd, 2.07 wt%; Zr, 0.6 wt%; Mg, balance; was used in the present study. The as-received 25 mm thick cast plate was machined into rectangular plates of 6 mm thickness each. These plates were friction stir processed in a temperature controlled atmosphere to achieve desired microstructure in the UFG regime (<1 μm). In order to control the heat generated by the tool and the plate surface interaction during FSP, an external cooling setup was used. A copper backing plate was used as a heat sink to absorb the heat produced during the process. Simultaneously, compressed air was also sprayed over-the-top surface of the processed zone to cool the samples quickly. The FSP was carried out in two passes with a tool rotation rate of 800 rpm and 600 rpm respectively at a traverse speed of 100 mm/min and tool tilt angle of 1.5° by employing a custom designed FSP tool. The FSP tool geometry consists of a concave shoulder of diameter 12 mm, a cone threaded pin of 6 mm diameter at the root and height of 3.4 mm.

2.2. High-temperature tensile testing

Mini tensile samples of 3 mm gage length, 1 mm width, and 1.5 mm thickness were machined by wire cut EDM along the processing direction from the stirred zone. High-temperature tensile tests were conducted at various strain rates in the range of 5×10^{-4} – 1×10^{-2} s^{-1} and temperatures in the range of 300–450 °C. An Instron 650 micro-tensile testing machine with the high-temperature environmental chamber was employed to conduct the tensile experiments at high-temperatures. The accuracy of the furnace temperature was controlled at ± 1 °C during testing.

2.3. Microstructural characterization

The microstructural examination was conducted using light microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD). For light microscopy and SEM characterization, the samples were polished up to 1 μm diamond paste followed by etching in a freshly prepared picric acid solution containing 4.2 g picric acid, 10 ml acetic acid, 10 ml distilled water and 70 ml ethanol. Fractography study was carried out on the failed samples after superplastic deformation to study the cavitation behavior.

EBSD samples are prepared by polishing with colloidal silica suspension of 0.02 μm followed by ion-milling in an argon atmosphere. Ion milling was done on the Gatan™ PECS-682 system operated at 5.5 keV. EBSD samples were scanned in FE-SEM integrated with EDAX-OIM software operating at 15 kV, 10 mm of working distance, using a spot size of 6. EBSD data were collected from the top surface of the stirred zone for the non-deformed sample and near the fracture tip for the tensile deformed sample carried out at high temperatures.

For TEM characterization, 500 μm thin foils of friction stir processed samples are sliced from the stirred zone using precision low speed cutting machine and mechanically grounded to less than 80 μm. These thin foils are punched into 3 mm discs and were ion-thinned with an incident angle of 6° and beam energy of 5 eV in an argon environment. TEM was done in a Phillips-CM12 electron microscope operating at 120 kV.

3. Results

3.1. Microstructures of the as-received and UFG QE22 alloy

The microstructure of the as-received QE22 alloy is mainly composed of: (i) coarse equiaxed α-Mg grains (Fig. 1(A)) which is surrounded by thermally stable $Mg_{12}Nd$ eutectic particles decorated along the grain boundary and (ii) fine ternary phase