Materials Science & Engineering A 620 (2015) 233–240

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Improvement of high strain rate and room temperature superplasticity in Zn–22Al alloy by two-step equal-channel angular pressing

M. Demirtas [a], G. Purcek [b,*], H. Yanar [b], Z.J. Zhang [c], Z.F. Zhang [c]

[a] Department of Mechanical Engineering, Bayburt University, Bayburt 69000, Turkey
[b] Department of Mechanical Engineering, Karadeniz Technical University, Trabzon 61080, Turkey
[c] Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China

ARTICLE INFO

Article history:
Received 11 July 2014
Received in revised form
29 September 2014
Accepted 30 September 2014
Available online 7 October 2014

Keywords:
Zn–22Al alloy
High strain rate superplasticity
Ultrafine-grained materials

ABSTRACT

The Zn–22Al alloy was subjected to equal-channel angular pressing (ECAP) to improve its high strain rate (HSR) superplasticity at room temperature (RT). A well-designed two-step ECAP process formed an ultrafine-grained (UFG) microstructure with an average grain size of 200 nm as the lowest one obtained so far after ECAP processing of this alloy. Also, agglomerate- and texture-free microstructure with UFG Al-rich α- and Zn-rich η-grains separated mostly by high-angle grain boundaries (HAGBs) was produced by this process. The maximum RT elongation was achieved to be 400% with a strain rate sensitivity of 0.30 at a very high strain rate of 5×10^{-2} s^{-1} after the two-step ECAP process. This elongation value is the highest one obtained at RT and at all strain rates for this alloy up to now. The current results demonstrate that such an improvement in superplasticity of Zn–22Al alloy after the two-step ECAP process can enhance its applications where RT and HSR superplasticity are strongly needed.

1. Introduction

Superplasticity is known to be the ability of some polycrystalline materials to exhibit very high elongation without necking prior to fracture as they are subjected to tension at temperatures above 0.5 Tm (Tm is the melting point of the material in Kelvin) and at low strain rates in-between 10^{-5}–10^{-3} s^{-1} [1,2]. It is also well known that superplasticity is obtained only if the grain size is decreased down to micron levels (typically less than 10 μm) due to the requirement of grain boundary sliding (GBS) [2]. However, some engineering applications require high or very high strain rate superplasticity at the possible lowest temperatures. Thus, great attention is being focused on developing materials or processing procedures for achieving the superplasticity for such applications.

Recent studies have shown that decreasing the grain size down to sub-micron level (ultrafine-grained (UFG) level) increases the strain rates and decreases the temperatures at which superplasticity occurs [1,3]. Experimental results have provided very clear demonstration on the formation of both high strain rate (HSR) and low temperature superplasticity in UFG materials where high strain rates refer to the tensile testing of samples at or above 10^{-2} s^{-1} and low temperatures refer to tensile testing at temperatures below 0.5 Tm. Therefore, it can be possible to achieve superplasticity at strain rates significantly faster than those associated with conventional superplasticity after UFG formation in superplastic materials. It has been well established that severe plastic deformation (SPD) is an effective way to produce UFG materials. Among the SPD techniques, high pressure torsion (HPT) and equal-channel angular pressing/extrusion (ECAP/E) are the most commonly used ones, and there are recent review papers tabulating the results for superplasticity after both HPT [4] and ECAP [1]. The potential of UFG materials produced by ECAP to obtain a superplastic capability at very high strain rates and relatively low temperatures was first suggested several years ago, and many studies have been performed to date in this area. Some of the alloys used in those studies can be summarized to be Al-based alloys [5–7], Mg-based alloys [8,9], Zn-based alloys (mainly Zn–22%Al alloy) [10–12], and copper-based alloys [13,14].

On the other hand, it has been shown that some alloys can show HSR superplastic behavior even at room temperature (RT) after UFG formation in their microstructures. Among them, eutectoid Zn–22Al alloy with micro-duplex phase structure is one of the most commonly used one for this purpose [3,11,12,15–20]. For producing UFG Zn–22Al alloy, some specific methods such as thermo-mechanical control process (TMCP) [3,15,16], ECAP [11,12,17–19], friction stir processing (FSP) [20], torsional straining (TS) [21,22], and cross-channel angular extrusion (CCAE) [23] have been utilized. According to the available studies, the maximum RT

* Corresponding author. Tel.: +90 4623772941; fax: +90 4623253336.
E-mail address: purcek@ktu.edu.tr (G. Purcek).

http://dx.doi.org/10.1016/j.msea.2014.09.114
0921-5093/

elongation for Zn–22Al was 340% at low strain rate of $10^{-5}\,s^{-1}$ after TMCP [3,15]. As the strain rate increased to $10^{-2}\,s^{-1}$, the elongation decreased to 180%. As seen, a limited improvement in HSR superplasticity at RT was achieved after this process due to the limited grain refinement (800 nm). After the one-step ECAP process, the maximum RT elongation was around 335% at a strain rate of $4\times10^{-3}\,s^{-1}$ [11]. Above this strain rate, the superplastic elongation dropped suddenly. Elongations of about 310% and 220% were achieved at higher strain rates of $10^{-2}\,s^{-1}$ and $10^{-1}\,s^{-1}$, respectively [11]. The mean grain size was 550 nm after this process. It was found that the FSP had no considerable effect on the superplasticity of Zn–22Al alloy. While RT superplasticity was not affected by grain refinement via FSP at low strain rates, the superplastic elongation increased from ~100% to ~150% at a high strain rate of $10^{-2}\,s^{-1}$ after this process [20]. The effects of the TS and CCAE processes on the superplasticity of Zn–22Al alloy were examined in high temperature regime [22,23]. The TS process decreased the grain size of Zn–22Al alloy from 1400 nm to 350 nm. This formation resulted in an improved HSR superplasticity at 200 °C with an elongation of 1800% at $10^{-1}\,s^{-1}$ strain rate [22]. The CCAE refined the microstructure with a 300 nm grain size. After this process, the maximum elongation at 200 °C was achieved to be 1092% at $5\times10^{-3}\,s^{-1}$ [23]. All these results are very important for improving HSR superplasticity of Zn–22Al alloy at RT. However, there is still a demand for improving HSR superplasticity of this alloy especially at RT for enhancing its applications where such superplastic behavior is strongly needed. Therefore, the purpose of this study is to improve RT superplasticity of Zn–22Al alloy at HSRs especially above $10^{-2}\,s^{-1}$ by utilizing a well-designed ECAP process. For this purpose, a two-step ECAP process was applied to develop a UFG microstructure with the lowest equiaxed grain size giving the highest RT superplasticity at the possible highest strain rates.

2. Experimental procedure

Proper amounts of Zn with 99.9% purity and Al with 99.9% purity were melted in a graphite crucible and cast into a cone-shaped steel mold for producing the Zn–22 wt%Al alloy. The as-cast ingots were homogenized at 375 °C for 24 h. Billets with dimensions of 13 mm × 13 mm × 20 mm were machined from those ingots for ECAP processing, and then homogenized at 375 °C for 48 h for the second time and quenched in order to obtain an initial microstructure with equiaxed Zn-rich η-phase and Al-rich α-phase. A well-designed two-step ECAP processing procedure was applied to the quenched Zn–22Al billets. In this process, the billets were extruded using an ECAP die with a sharp 90° channel cross-section angle at a rate of $1\,mm\,s^{-1}$. For the first step, the billets were processed up to four passes at 350 °C which is above the eutectoid line of the alloy. For the second step, four more passes were also applied to these billets at RT. The billets were processed by ECAP via route-Bc where they are rotated 90° in the same direction around their longitudinal axis between successive passes. This route was especially chosen to generate a fine equiaxed microstructure with a high volume fraction of high-angle grain boundaries (HAGBs) [24].

The microstructure of the alloy was observed by transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) using an FEI Tecnai F20 microscope, operated at a nominal voltage of 200 kV. A detailed investigation of UFG microstructure in the qualitative and quantitative manners after the two-step ECAP process was also undertaken by the electron backscattering diffraction (EBSD) technique in combination with a LEO Supra 35 field emission scanning electron microscope (SEM) operated at a nominal voltage of 20 kV. Pole figures were determined by the SEM-EBSD method to evaluate the texture formation after the process. All these investigations were undertaken on the cross-sections perpendicular to the extrusion direction of the billets (Fig. 1).

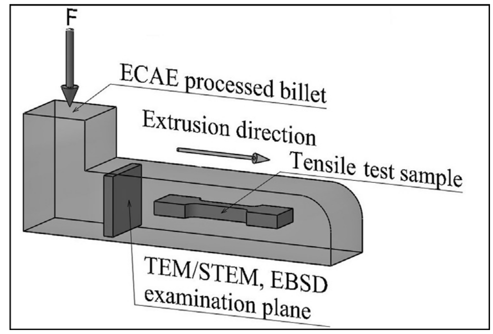

Fig. 1. A schematic representation describing the orientations of the tensile test and microstructural examination samples in ECAP-processed billets.

For the evolution of HSR superplasticity at RT, tensile tests were performed at different strain rates ranging from $10^{-3}\,s^{-1}$ to $10^{0}\,s^{-1}$ at about 25 °C. Dog-bone shaped samples with dimensions of 2 mm × 3 mm × 21 mm were machined from the processed billets using wire electro-discharge machining (wire-EDM) for tensile tests. The samples were cut from the ECAP-processed billets where their tensile axis aligned with the direction of extrusion (Fig. 1). All the samples were polished before the tensile tests in order to eliminate the effect of oxidation and any micro-cracks on the surfaces coming from wire-EDM cutting. Tension tests were performed using an Instron-3382 electro-mechanical load frame with a video type extensometer. For each case, at least three experiments were performed on companion samples to check the repeatability of the results. In order to characterize the flow behavior and determine the deformation mechanism of the alloy during tension in the optimum superplastic region, the surface appearances of the samples which had been strained up to 100% and 130% at a strain rate of $5\times10^{-2}\,s^{-1}$ were also examined using the SEM. Before straining, the surfaces of the samples were ground and then polished to reveal the structural details.

3. Results

3.1. Microstructure

TEM micrographs showing the microstructures of Zn–22Al alloy after the two-step ECAP process are shown in Fig. 2. In these micrographs, the bright and dark contrasts correspond to the Al-rich α-phase and Zn-rich η-phase, respectively. It is clearly seen from Fig. 2 that the two-step ECAP process formed a UFG microstructure with well-defined equiaxed grains in both α- and η-phases. No dislocation formation was seen after ECAP according to the TEM micrographs as expected (Fig. 2). This process was especially designed to produce a microstructure with the possible lowest grain size and to obtain the possible highest RT elongation at very high strain rates. Therefore, more investigation was also undertaken in order to examine this microstructure in details. Fig. 3(a) and (b) shows the STEM micrographs indicating well-defined UFG morphology of Zn–22Al alloy. In addition, energy dispersive X-ray (EDX) analysis spectrums are also given in the inset of the micrograph for identifying the compositions of each phase (Fig. 3(b)). From these spectrums, in contrast to the TEM micrographs, the bright and dark contrasts correspond to the Zn-rich η-phase and Al-rich α-phase, respectively. STEM micrographs