





# Improvement of room-temperature superplasticity in Zn–22 wt.%Al alloy

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#### **Abstract**

Zn-22 wt.%Al alloy with sub-micrometer-grained microstructure was prepared by three different processing routes: (1) solid solution treatment + quenching + 8 passes equal channel angular pressing (ECAP) at room temperature (RT), and (3) Route 1+cryo-rolling at 203 K (with a reduction of 75%). The samples were tested in tension at strain rates range from  $1 \times 10^{-4}$  to  $1 \times 10^{0}$  s<sup>-1</sup> at room temperature. All the samples processed through these three routes exhibit good superplasticity at room temperature. At the strain rate of  $4 \times 10^{-3}$  s<sup>-1</sup>, samples processed via Routes 2 and 3 reach a high elongation of 335 and 315%, respectively. At a higher strain rate of  $1 \times 10^{-1}$  s<sup>-1</sup>, the elongation of samples processed by these two routes is still higher than 220%. Besides the high elongation, at strain rate of  $4 \times 10^{-2}$  s<sup>-1</sup>, a high value of strain-rate sensitivity ( $\sim$ 0.35) was observed in samples processed by Route 2. A room-temperature grain growth during ECAP was also observed in the alloy. © 2007 Elsevier B.V. All rights reserved.

Keywords: Room-temperature superplasticity; Zn-22 wt.%Al; ECAP; Cryo-rolling; Strain-rate sensitivity

#### 1. Introduction

High tensile ductility may be observed in some metals when the grain size is very small, typically less than 10  $\mu$ m, and when the testing is conducted at a relatively high homologous temperature of the order of at least 0.5  $T_{\rm m}$ , where the  $T_{\rm m}$  is the melting point of the material in Kelvin. Experiments show that superplastic deformation is achieved over a limited range of strain rates of  $10^{-5}$  to  $10^{-1}\,{\rm s}^{-1}$ . Maintaining the superplastic property at lower temperature over the higher strain rates to achieve room-temperature (RT) superplasticity is especially important when attempts are made to extend the application of superplasticity.

Grain boundary sliding is the dominant flow mechanism in superplasticity [1–4] and there is experimental evidence showing that a refinement in grain size leads to room-temperature superplasticity over a relatively high strain rate: for example, a refinement of grain size to 2.5 µm in the Zn–22 wt.%Al eutectoid alloy achieved the elongation of 188% at room temperature [5],

and a refinement of grain size to 0.7– $0.9\,\mu m$  by equal channel angular pressing (ECAP) in the same alloy led to a superplastic elongation of 280% [6]. These results showed the strong dependence of room-temperature superplasticity on grain size in the alloy.

Although there are many efforts to achieve room-temperature superplasticity in Zn–22 wt.%Al alloy [5–12], most of these work merely obtain an elongation value within 200%, and a strain-rate sensitivity less or close to  $\sim$ 0.2. However, an elongation value over 300% and a strain-rate sensitivity over 0.3 are usually thought to be essential to indicate the characteristic of superplasticity. It is worthy to optimize the effect of processings, such as equal channel angular pressing, to search the potential of obtaining superplastic elongation over 300% at room temperature.

Different combinations of heat treatment (solid solution treatment, aging, etc.) and deformation processings (ECAP, cryo-rolling, etc.) are used in the present work to achieve better control of ultrafine-grained microstructure in Zn–22 wt.%Al eutectoid alloy. The room-temperature superplasticity and microstructure are characterized by testing in tension and transmission electron microscopy (TEM), an exceeding superplastic elongation 300% at room temperature is obtained with grain size

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Table 1 Three different processing routes

| Number  | Treatment                               |
|---------|---|
| Route 1 | Solid solution treated at 633 K for     |
|         | 24 h + quenching + aging at RT for 72 h |
| Route 2 | Solid solution treated at 633 K for     |
|         | 24 h + quenching + ECAP 8 passes at RT  |
| Route 3 | Route 1 + cryo-rolling at 203 K         |

from  $\sim$ 0.25 to  $\sim$ 0.55  $\mu$ m. Experimental results show that different processing routes have great influence on microstructure and superplastic deformation behavior.

# 2. Experimental procedure

A commercial Zn–22 wt.%Al binary alloy with an asreceived grain size of  $\sim\!10~\mu m$  was used in present investigation. Alloys were prepared for three different processing routes, which are summarized in Table 1. Samples for ECAP were prepared in the form of rods with diameters of 30 mm and total lengths of 160 mm, which were pressed at room temperature using an ECAP facility having  $\phi=90^\circ,\,\varphi=22.5^\circ.$ 

The microstructure of samples before and after testing in tension were examined using TEM after cutting samples, polishing to a thickness of 100–120  $\mu m$ , then punching out small disks with a diameter of 3 mm. A twin-jet electro-polishing unit was used with a solution of 6% HClO<sub>4</sub>, 94% C<sub>2</sub>H<sub>5</sub>OH and samples were thinned to perforation at a temperature of  $\sim\!253\,K$ . Specimens were examined in a JEM-2000EX transmission electron microscope.

For testing in tension of the samples following the three processing routes, they were cut into tensile specimens with gauge lengths of 5 mm and cross-sections of 2 mm  $\times$  3 mm. They were tested in tension to failure using an Instron-type machine oper-

ating at room temperature covering a range of initial strain rates from  $1 \times 10^{-4}$  to  $1 \times 10^{0}$  s<sup>-1</sup>.

# 3. Experimental results

#### 3.1. Microstructure characteristics

The typical microstructure of samples processed by Routes 1–3 are shown in Fig. 1. After quenching and aging, the microstructure consists of equiaxed grains with an aspect ratio close to  $\sim\!1$ , and with diameter from  $\sim\!0.15$  to  $\sim\!0.7~\mu m$ . The mean grain size is  $\sim\!0.35~\mu m$ . Also we calculated the distribution of grain size by measuring more than one hundred grains and 70% of which are in size of less than 0.2  $\mu m$ . Close inspection shows that the grains contain relatively few dislocations and the grain boundaries are reasonably smooth, as shown in Fig. 1a.

After 8 passes of ECAP following solid solution treatment and quenching (Route 2), the grains are more equiaxed with an aspect ratio close to  $\sim 1$ . Their boundaries are straighter than in Route 1, and tend to be polygonal in shape. The largest grain size has been estimated to be  $\sim 0.9 \, \mu m$ , and a majority of grain sizes cover the range of  $0.5-0.6 \, \mu m$ , with an average grain size of  $\sim 0.55 \, \mu m$ , as shown in Fig. 1b.

Fig. 1c shows the microstructure of sample obtained by Route 3, with an average grain size of  $0.25~\mu m$ . Elongated grains along the rolling direction is obvious in both Al-rich and Zn-rich phases, and the aspect ratio of grains is close to 2.6.

### 3.2. Mechanical properties

All the specimens were tested in tension to failure at different initial strain rates. The dependence of elongation to failure  $(\Delta L/L_0)$ , flow stress  $(\sigma)$  and strain-rate sensitivity (m) upon initial strain rates, are summarized in Figs. 2–4. The samples processed by Routes 2 and 3 have a maximum elongation to

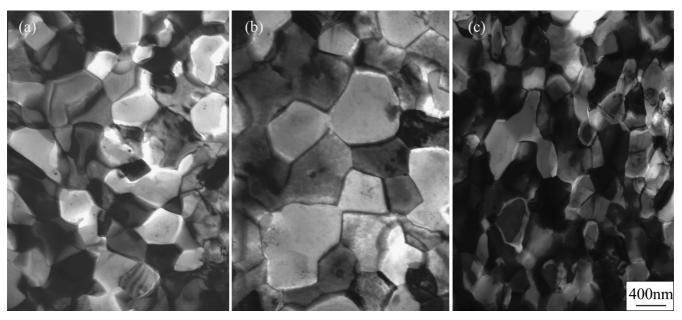


Fig. 1. Typical microstructure observed in samples after processing by three different routes: (a) Route 1; (b) Route 2; (c) Route 3.

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