IOP Conf. Series: Materials Science and Engineering **194** (2017) 012033 doi:10.1088/1757-899X/194/1/012033

Optimization of RT superplasticity of UFG Zn-22Al alloy by applying ECAP at different temperatures and phase regions

M Demirtas[1,2], H Yanar[2], G Purcek[2]

[1]Bayburt University, Faculty of Engineering, Department of Mechanical Engineering, 69000, Bayburt, Turkey
[2]Karadeniz Technical University, Faculty of Engineering, Department of Mechanical Engineering, 61080, Trabzon, Turkey

E-mail : purcek@ktu.edu.tr

Abstract. Zn–22Al alloy was subjected to either one-step or two-step equal channel pressing (ECAP) to investigate the effect of processing temperature on its microstructure and room temperature (RT) superplasticity. In one-step ECAP processes, 4 passes ECAP were applied to the alloy at different temperatures: RT, 100°C and 250°C in two-phase region below eutectoid temperature and 350°C in single-phase region above eutectoid temperature. In two-step ECAP processes, one-step ECAP-processed samples were subjected to four more passes ECAP at RT. Considering the one-step ECAP processing, RT superplasticity increased with decreasing ECAP temperature as expected, and the highest RT superplasticity was achieved as 350% after 4 passes ECAP at RT. On the other hand, application of 4 more passes ECAP at RT to the sample showing the lowest superplastic elongation after one-step ECAP (the sample processed at 350°C) resulted in the maximum RT elongation of 400% at a high strain rate of $5x10^{-2}$ s^{-1}. These results suggest that first step temperature of two-step ECAP process is needed to increase above the eutectoid point of Zn-22Al alloy to achieve high RT superplasticity. These results were attributed to the changes in microstructure inside the single-phase and two-phase regions during the processes.

1. Introduction

Superplastic behavior can be explained as the high neck-free tensile elongation to failure which occurs at some polycrystalline materials. High tensile test temperature (about 0.5Tm where Tm is the absolute melting point of the material), low strain rate (between $1x10^{-5}$ s^{-1} and $1x10^{-3}$ s^{-1}) and small grain size (typically below than 10 μm) are the main requirements that should be fulfilled in order to achieve superplasticity [1, 2]. Among these requirements, grain size can be considered as the most important one since any change in grain size also affects two other requirements. Decreasing grain size decreases the temperature and increases strain rate at which superplasticity are achieved [3, 4]. Therefore, grain refinement techniques have been among the main interests of scientist and engineers who deal with the superplastic behavior of metals.

In order to achieve fine grained (FG) and/or ultrafine grained (UFG) microstructures, some thermal and thermomechanical processes have been applied to superplastic materials [5-18]. Considering the thermomechanical processes, severe plastic deformation (SPD) techniques have been developed and performed successfully [5, 9-18] besides the conventional plastic deformation processes like rolling, extrusion and forging. Furthermore, SPD techniques have been found to be more effective to achieve smaller grain size and thus higher superplastic elongation [14] comparing to the conventional grain refinement techniques. Equal channel angular pressing (ECAP) [5, 9-14], friction stir processing (FSP) [15], torsional straining (TS) [16,17] and cross-channel angular extrusion (CCAE) [18] are the main SPD techniques which have been used for grain refinement in superplastic materials. Among them, ECAP is the most commonly used one and it has been applied to many different superplastic materials in order to achieve FG and UFG microstructures. For this purpose Zn-22Al alloy as a model superplastic material is one of the most commonly studied superplastic alloy and ECAP has been applied to this alloy at different temperatures as a grain refinement tool [5, 9-14].

Effects of some ECAP parameters on the final microstructure and mechanical properties of the materials have been studied [19] including processing route, numbers of passes, pressing speed, back pressure and pressing temperature. Pressing temperature is considered as a key factor and it has been shown that lower pressing temperature leads to lower final grain size [19]. On the other hand, two-step ECAP in which 4 passes were applied to Zn-22Al alloy at 350 ºC followed by 4 more passes at RT (8 total passes) resulted in higher superplastic elongation [14] compared to 8 passes ECAP performed at room temperature (RT) [5]. Thus, two step ECAP seems to be more suitable to achieve higher superplastic elongations than ECAP in which all passes are performed at low temperature. Regarding the dependency of final grain size of superplastic materials to the ECAP temperature, it will be beneficial to determine whether it is possible to achieve higher superplastic elongations by decreasing the first step ECAP temperature (like performing first step ECAP at 100 ºC or 250 ºC) of two-step ECAP processes or not. Therefore, the main purpose of this study is to analyze the effect of first step temperature of two-step ECAP on the RT superplasticity of Zn-22Al alloy.

2. Experimental procedure

As-cast Zn-22Al ingot was homogenized at 375 ºC for 24 h and billets with dimensions of 13 x 13 x 130 mm^3 were machined from the ingot for the subsequent ECAP. After then, the billets were homogenized at 375 ºC for 48 h for the second time and quenched into ice-water. ECAP was performed as either one-step or a two-step processes. In the one-step ECAP, each billet was subjected to 4 passes at RT, 100 ºC and 250 ºC in the two phase region below the eutectoid temperature and at 350 ºC in the single phase region above the eutectoid transformation temperature of the alloy. In the two-step ECAP, 4 more passes were applied to the billets at RT followed by the 4 passes performed in the first step. ECAP processes were conducted at $1mm.s^{-1}$ pressing speed using route Bc where the billets were rotated along their longitudinal axis among each passes.

Scanning electron microscopy (SEM) was utilized for the microstructural examination after the applied processes. For this purpose, SEM examination samples were cut from the ECAP-processed billets as so examination plane was perpendicular to the extrusion direction (ED) of the billets using wire electro-discharge machining (wire-EDM). The samples were ground, polished and etched in a solution containing 5 g CrO_3, 0.5 g Na_2SO_4 and 100 ml H_2O. SEM was conducted using a JEOL-6400 microscope.

In order to evaluate the RT superplasticity after the applied processes, tensile tests were performed at strain rates ranging between $1x10^{-3}$ and $1x10^{0}$ s^{-1}. Dog bone shaped test samples having 2 mm x 3 mm x 5 mm gauge section dimensions were extracted from the billets with their tensile axis aligned with the ED. All tests were repeated at least three times in order to confirm the validity of the tensile test results and mean of these tests results are given in the manuscript.

3. Results and discussion

SEM micrographs showing the microstructures of the alloy after the applied processes are given in figure 1. In these micrographs, the bright and dark contrast correspond to Zn-rich η- and Al-rich α-phases, respectively. 4 passes ECAP at RT resulted in a UFG microstructure with 350 nm grain sizes (Figure 1(a)-(b)). In one-step ECAP processes, increasing the ECAP temperature up to 250 ºC also increased the final grain size, and grain sizes after 4 passes ECAP performed at 100 ºC and 250 ºC were measured to be 500 nm ((Figure 1(c)-(d))) and 1 μm (Figure 1(e)-(f)), respectively. On the other hand, application of ECAP above the eutectoid temperature brought about more refined microstructure with 250 nm grain size (Figure 1(g)-(h)). However, the microstructure consists of some regions with lamellar structure (LS) after that process (Figure 1(h)).

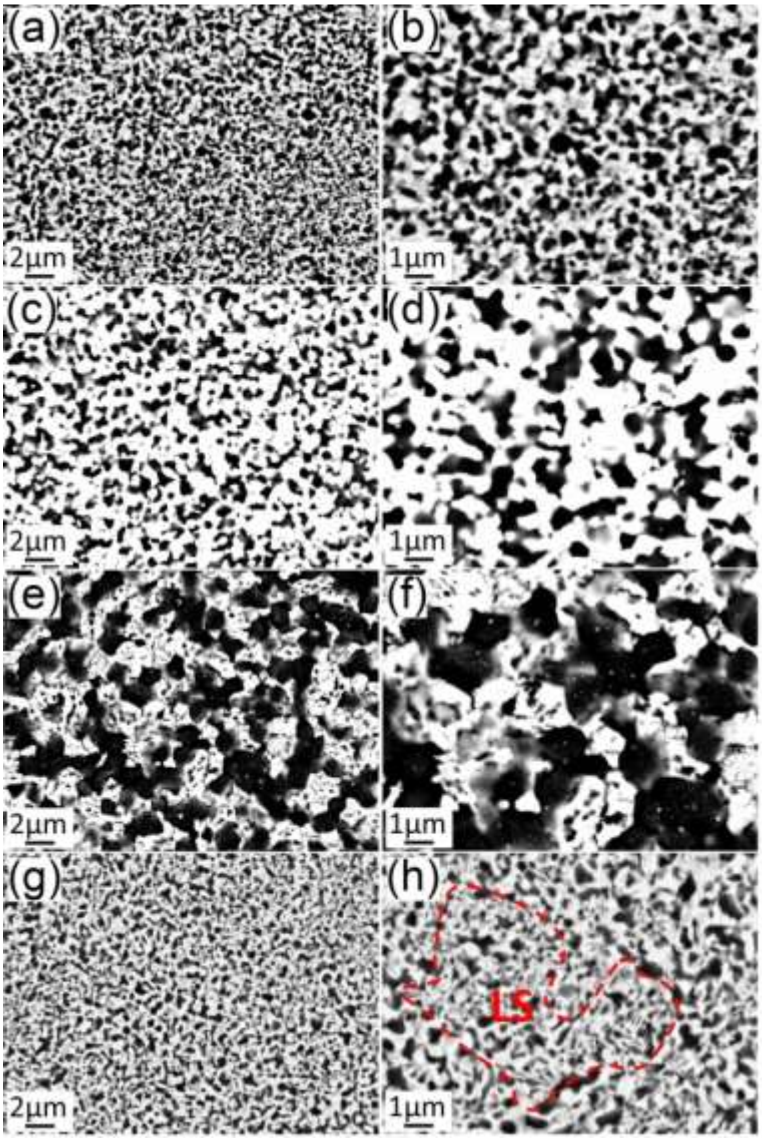

Figure 1. SEM micrographs showing the microstructures after one-step ECAP performed at: (a)-(b) RT, (c)-(d) 100 ºC (e)-(f) 250 ºC and (g)-(h) 350 ºC.

Application of four more passes ECAP at RT to the one-step ECAP-processed samples resulted in more refined microstructures compared to one-step ECAP processes. 8 total passes at RT brought about 250 nm grain sized microstructure (Figure 2(a)-(b)). Grain sizes after 4 passes ECAP at RT following one-step ECAP at 100 ºC and 250 ºC were measured as 400 nm (Figure 2(c)-(d)) and 700 nm (Figure 2(e)-(f)), respectively. Lamellar structure formed after 4 passes ECAP at 350 ºC was completely eliminated by 4 more passes ECAP at RT (Figure 2(g)-(h)). Lamellar-free UFG microstructure with 200 nm grain size was achieved instead [14].

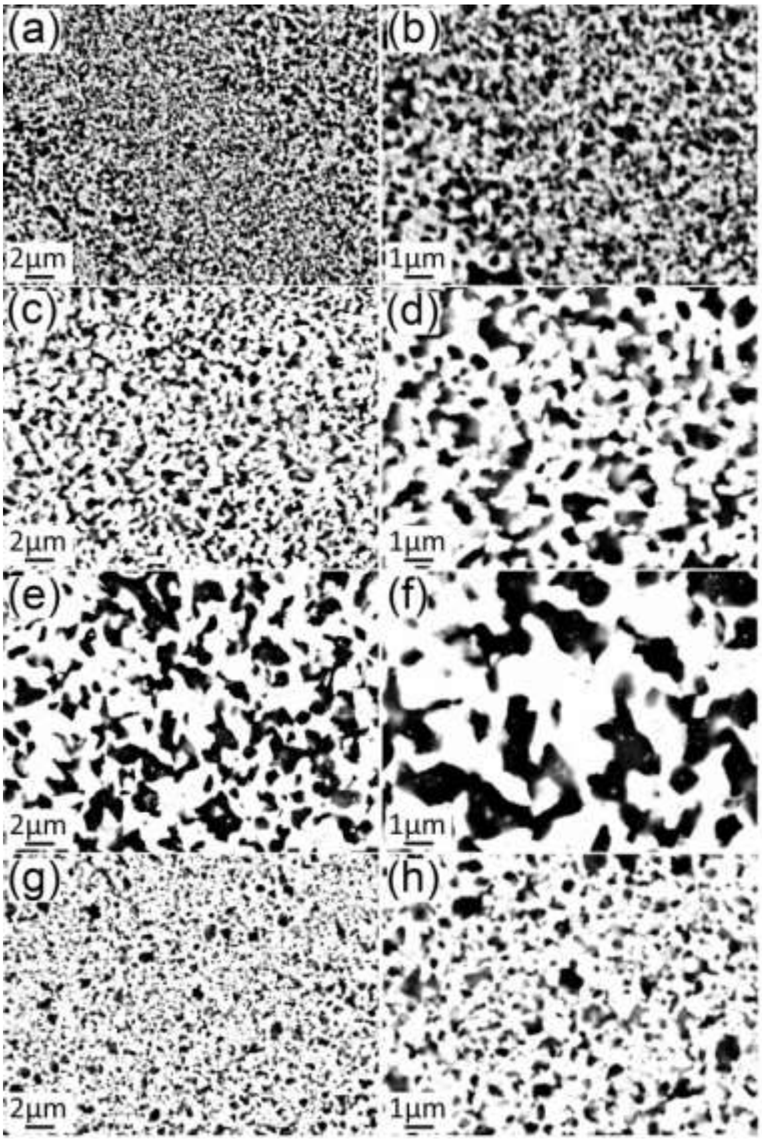

Figure 2. SEM micrographs showing the microstructures after two-step ECAP processes in which first step was performed at: (a)-(b) RT, (c)-(d) 100 ºC (e)-(f) 250 ºC and (g)-(h) 350 ºC.

It was stated previously that decreasing pressing temperature of ECAP resulted in finer grain sizes [19]. The final grain sizes obtained after one-step ECAP processes are in a good agreement with that conclusion except for the 4 passes ECAP applied at 350 ºC; i.e. below the eutectoid temperature the smallest grain size was achieved after 4 passes ECAP at RT. Regarding all one-step ECAP processes, on the other hand, although 350 ºC is the highest process temperature, the smallest grain size was achieved after 4 passes ECAP at 350 ºC. Considering ECAP temperatures of RT, 100 ºC and 250 ºC, the alloy was processed at two phase region, and grain refinement occurred due to the only ECAP. However, eutectoid transformation occurred at 350 ºC before the ECAP process, and ECAP performed in one phase region in that process. In this process, the ECAP billets were quenched between each passes. It is known that, quenching above the eutectoid temperature results in refined microstructure in Zn-22Al alloy [20]. Thus, grain refinement effects of both ECAP and quenching are combined, and the smallest grain size was achieved after 4 passes ECAP at 350 ºC.

Considering the grain sizes achieved after the applied processes, it can be concluded that decreasing ECAP temperature below the eutectoid temperature also decreases final grain size. However, processing the alloy above the eutectoid temperature results in more refined microstructure with some lamellar structure which is not suitable for achieving high superplastic elongation [20]. Thus, in one-step ECAP processes it is beneficial to keep ECAP temperature as low as possible for desired microstructure for superplasticity. On the other hand, in two-step ECAP, the smallest grain size was achieved after 4 passes ECAP at 350 ºC followed by 4 more passes at RT. Therefore the alloy should be processed above the eutectoid temperature in the first step of two-step ECAP processes in order to achieve more refined microstructure.

The RT superplastic elongations after the applied processes with respect to the initial strain rates are shown in figure 3. Considering the results of one-step ECAP processes, the highest RT elongation was achieved after 4 passes ECAP at RT as 350% at a strain rate of $1x10^{-1}$ s^{-1} (Figure 3(a)). 4 passes ECAP at 350 ºC, 250 ºC and 100 ºC resulted in maximum elongations of 110%, 195% and 315%, respectively (Figure 3(a)). Further grain refinement in two-step ECAP processes by means of 4 more passes ECAP at RT increased elongation to failure comparing to all one-step ECAP processes. In general, the sample subjected to 4 passes ECAP at 350 ºC + 4 passes ECAP at RT reflected to the highest RT elongation to failure as 400% at a strain rate of $5x10^{-2}$ s^{-1} (Figure 3(b)) [14]. 8 total ECAP passes at RT also resulted in high superplastic elongation of 375% [1] at $1x10^{-1}$ s^{-1}(Figure 3(b)

Decreasing grain size increases the maximum superplastic elongations in superplastic materials due to the effective grain boundary sliding (GBS) which occurs as the main deformation mechanism at more grain boundaries [3, 4]. Considering the maximum elongations after one-step ECAP processes, the results are consistent with this conclusion except for the maximum elongation obtained after 4 passes ECAP at 350 ºC. Although the alloy has the smallest grain size after 4 passes ECAP at 350 ºC among all one-step ECAP-processed samples, the lowest superplastic elongation was achieved after this process. Similar observation was also achieved in Zn-22Al alloy in [20]. It was shown that, lamellar structure inside the microstructure makes difficult the accommodation process of GBS by intragranular dislocation slip process and causes stress concentration especially near the lamellar structure. Thus, in the early stage of superplastic deformation (about 50% superplastic elongation) some micro-cracks nucleate near the lamellar structure and premature failure occurs [20]. Similarly, the unexpected low superplastic elongation after 4 passes ECAP at 350 ºC can be attributed to the partially lamellar structure which causes stress concentration and thus premature failure.

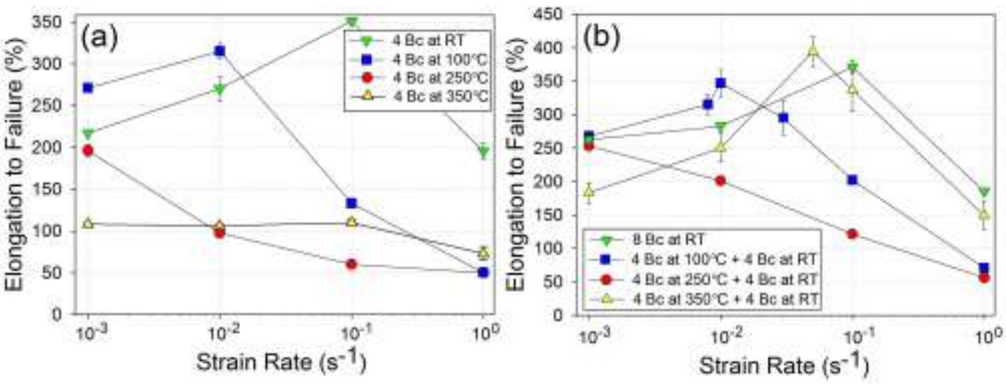

Figure 3. Variations of elongation to failure with the initial strain rates after: (a) one-step ECAP, and (b) two-step ECAP processes.

4. Conclusions

In this study, the effects of one-step and two-step ECAP temperatures on the final grain size and RT superplasticity of Zn-22Al alloy were investigated. The main findings and conclusions can be listed as below.

1. Decreasing ECAP temperature below the eutectoid temperature also decreases final grain size. However, processing the alloy above the eutectoid temperature results in more refined microstructure with some lamellar structure.

2. In one-step ECAP processes, it is beneficial to keep ECAP temperature as low as possible. On the other hand, the alloy should be processed above the eutectoid temperature in the first step of two-step ECAP processes in order to achieve more refined microstructure.
3. In one step ECAP, 4 passes at 350 ºC resulted in the lowest superplastic elongation although it brought about the lowest grain size. This unexpected low superplastic elongation was attributed to the partially lamellar structure which causes stress concentration and thus premature failure. The maximum RT elongation was achieved to be 400% at a strain rate of $5x10^{-2}$ s^{-1} after two-step ECAP process which resulted the lowest grain size among all applied processes.

Acknowledgments
This research was supported by Scientific Research Projects of Karadeniz Technical University, Turkey, under Grant no10501.

References

[1] Kawasaki M and Langdon T.G 2014 *J. Mater. Sci.* **49** 6487.
[2] Kaibyshev O.A 1992 *Superplasticity of Alloys Intermetallides and Ceramics*, (Berlin: Springer-Verlag).
[3] Langdon T.G 1994 *Mater. Sci. Eng. A* **174** 225.
[4] Mayo M.J 1997 *Nanost. Mater.* **9** 717.
[5] Xia S.H, Wang J, Wang J.T and Liu J.Q 2008 *Mater. Sci. Eng. A* **493** 111-115.
[6] Tanaka T, Makii K, Ueda H, Kushibe A, Kohzu M and Higashi K 2003 *Inter. J. Mech. Sci.* **45** 1599.
[7] Tanaka T, Makii K, Kushibe A and Higashi K 2002 *Mater. Trans.* **43** 2449.
[8] Tanaka T, Makii K, Kushibe A, Kohzu M and Higashi K 2003 *Scr. Mater.* **49** 361.
[9] Tanaka T, Watanabe H and Higashi K 2003 *Mater. Trans.* **44** 1891.
[10] Tanaka T and Higashi K 2004 *Mater. Trans.* **45** 1261
[11] Kumar P, Xu C and Langdon T.G 2006 *Mater. Sci. Eng. A* **429** 324.
[12] Huang Y and Langdon T.G 2002 *J. Mater. Sci.* **37** 4993.
[13] Yang C.F, Pan J.H and Chuang M.C 2008 *J. Mater. Sci.* **43** 6260.
[14] Demirtas M, Purcek G, Yanar H, Zhang Z.J and Zhang Z.F 2014 *Mater. Sci. Eng. A* **620** 233.
[15] Hirata T, Tanaka T, Chung S.W, Takigawa Y and Higashi K 2007 *Scr. Mater.* **56** 477.
[16] Furukawa M, Horita Z, Nemoto M, Valiev R.Z and Langdon T.G 1996 *J. Mater. Res.* **11** 2128.
[17] Kawasaki M and Langdon T.G 2011 *Mater. Sci. Eng. A* **528** 6140.
[18] Chou C.Y, Lee S.L, Lin J.C and Hsu C.M 2007 *Scr. Mater.* **57** 972.
[19] Valiev R.Z and Langdon T.G 2006 *Prog. Mater. Sci.* **51** 881.
[20] Demirtas M, Purcek G, Yanar H, Zhang Z.J and Zhang Z.F 2016 *J. Alloy. Compd.* **663** 775.