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INVESTIGATING MICROSTRUCTURES AND HIGH TEMPERATURE SUPERPLASTICITY BEHAVIOR OF MG-5(MASS%) SN ALLOY BY USING EQUAL CHANNEL ANGULAR EXTRUSION

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Key words: high temperature plasticity behaviour, Mg-5(mass%) Sn alloy, equal channel angular extrusion (ECAE), Mg_2Sn particles.

ABSTRACT

In this study, we investigated the microstructures and high temperature plasticity behaviour of an Mg-5(mass%) Sn alloy after equal channel angular extrusion (ECAE). These results show that the grain boundary of the as-cast Mg-5(mass%) Sn alloy contained continuous eutectic α-Mg + Mg_2Sn precipitates. After an ECAE process, the average grain size decreased from 147 μm to about 10 μm and the continuous eutectic α-Mg + Mg_2Sn particles were broken down, and those particles distributed uniformly. The maximum elongation was 550 % at high temperature 350°C with strain rate of 1×10^{-3} s^{-1}.

I. INTRODUCTION

Magnesium alloys are the lightest commercial structural alloys and have excellent specific strength and stiffness, and sounds better used in PC or portable information equipment (Kojima, 2001). Plasticity behavior has been proven to play a key role in the manufacturing of complicated parts in the industry. Fine structure plasticity of metals usually requires that the average grain size is distributed homogeneously below 10-15 μm without significant grain growth (Sherby, 1989; Nieh, 1997). High temperatures plasticity has found to exist in several magnesium alloys and their composites such as AZ31, AZ61, AZ91 and ZK60 (Bussiba, 2001; Hiroyuki, 2002; Tan, 2002; Hidetoshi, 2003; Wei, 2003; Yuichi, 2006). In order to increase plastic strain rate by reducing average grain size, several thermo-mechanical processes have been studied, such as extruding (Yuichi, 2006; Park, 2011; You, 2011), rolling (Tan, 2002; Wei, 2003) and equal channel angular extrusion (ECAE) (Hiroyuki, 2002; Yuichi, 2006).

Park et al. studied the extruded Mg-Sn based alloys (Park et al., 2011 and Park & You, 2011). These authors used the solution heat treatment (SHT) and indirect extrusion in a two-step process to produce ultrafine grains (1.5 μm). The results show maximum elongations achieving 670% and 900% at low temperature 200 °C with strain rate 1×10^{-4} s^{-1} due to the fine grain microstructure and the presence of small Mg_2Sn particles; You et al. studied the plasticity deformation mechanism of Mg-Sn based alloy at low temperatures (175°C and 200°C) (You et al., 2011). Therefore, we try to use ECAE process to reduce the size of grains and Mg_2Sn particles; then further evaluate the plasticity deformation mechanism of Mg-5(mass%) Sn alloy at high temperatures (250°C, 300°C and 350°C).

The Mg-5(mass%) Sn alloy was selected because earlier studies reported that the Mg-5(mass%) Sn alloy has good tensile properties and excellent creep resistance at high temperatures. Moreover, researchers investigated that the Mg_2Sn precipitates are very stable at high temperatures (Park, 2005; Chen, 2007; Liu, 2007; Kang, 2007; Wei, 2008; Wei, 2009; Tsai, 2012).

II. EXPERIMENTAL PROCEDURE

An alloy with a composition of Mg-5(mass%) Sn was prepared. Pure magnesium (99.95 mass%) and pure tin (99.98 mass%) were melted in a crucible under the protection of SF6 gas at 800°C. The melted mixture was stirred to ensure homogeneity. It was then held at 720°C for 30 minutes and finally cast into a steel mould that was preheated to 250°C. The cavity dimension of the mould was 300 mm × 70 mm × 60 mm. The ECAE was conducted by a die which is a block with two intersecting channels of identical cross-section with a 120° angled channel through the die via Bc processing route. Bc processing route means that the sample was removed from the die and then rotated by + 90° in the same direction between

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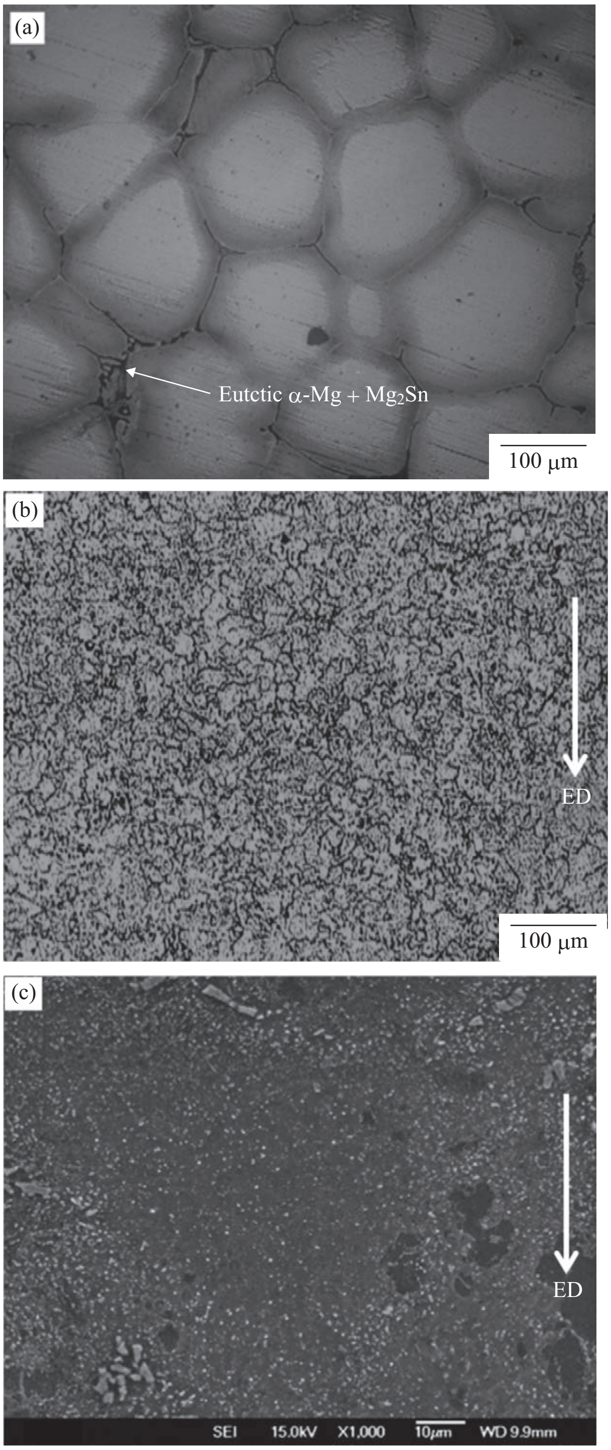

Fig. 1. **OM & SEM images of Mg-5(mass%) Sn alloy (a) OM image of as-cast state, (b) OM image of ECAE 6 passes, and (c) SEM image of ECAE 6 passes.**

each pass. The sample was then extruded through six passes at a temperature of 200°C with an extrusion rate of about 2 mm/min. The metallographic samples were sliced from the same place in each casting and ECAE processing sample.

They were polished and etched with a solution of 5 vol. % nitric acid + ethyl alcohol. The average grain size was measured by using the linear intercept method. The tensile tests were conducted at 250°C, 300°C and 350°C, on a tensile testing machine (Instron8500) with strain rates of 1×10^{-2}, 1×10^{-3} and 1×10^{-4} s^{-1}, respectively. The tensile specimens were cut from samples after ECAE processing by wire-electrode cutting and were made with a gauge length of 6 mm. The characteristics of grain coarsening at elevated temperature were investigated; the heat treatment was conducted at 350°C with different holding time (10, 30, 60, 360 and 720 min).

We used thermocouple and digital temperature controller to control and maintain the ECAE temperature. If the temperature changing was detected by thermocouple, the digital temperature controller could feedback and adjust the ECAE temperature immediately.

III. RESULTS AND DISCUSSION

1. Microstructure of the Mg-5(mass%) Sn Alloy

Figs. 1 (a) and (b) show the microstructure of the Mg-5 (mass%) Sn alloy in the as-cast state and in ECAE condition of six passes (N = 6). The arrow direction was Extrusion Direction (ED). The average grain size of Mg-5(mass%) Sn alloy is reduced from 147 μm (as-cast) to about 10 μm by ECAE six passes. It means that EACE process at 200°C favors dynamic recrystallization (DRX); the DRX leads to produce equiaxed grains with high concentration (Somjeet et al., 2009). Fig. 1(c) shows the SEM image of Mg-5(mass%) Sn alloy after ECAE six passes. The result shows that the fine Mg_2Sn particles are distributed very uniform in the Mg-5(mass%) Sn alloy. We suggest that those fine Mg_2Sn particles are formed by two reasons. First of all, the continuous Mg_2Sn precipitates are broken by ECAE six passes. Secondly, the Mg_2Sn particles are produced by dynamic precipitation during ECAE process. Further, those Mg_2Sn particles are very helpful to restrict grain growth after ECAE six passes at 200°C with low extrusion speed 2 mm/min. Early studies reported similar results (Matsubara, 2003; Wei, 2003; Miyahara, 2005); it was found that the fine $Mg_{17}Al_{12}$ particles formed via dynamic precipitation during extrusion, thus the grain boundary migration was resisted obviously by those fine $Mg_{17}Al_{12}$ particles during extrusion.

2. High Temperature Plasticity Behavior

All of the tensile elongations are shown in Fig. 2(a). The results show that superplasticity is achieved after the ECAE six passes with strain rate 1×10^{-4} s^{-1} at high temperatures (250°C, 300°C and 350°C). The maximum elongation of 550% is found at 350°C with strain rate 1×10^{-3} s^{-1}. It is larger than the elongation of 365% at 350°C with strain rate of 1×10^{-4} s^{-1}. Fig. 2(b) can explain this phenomenon; the results demonstrate the average grain size of Mg-5(mass%) Sn alloy after heat treatment at high temperature 350°C with different holding time (10, 30, 60, 360 and 720 min).The average grain size slightly grow from 10 μm to 14 μm at 350°C with holding

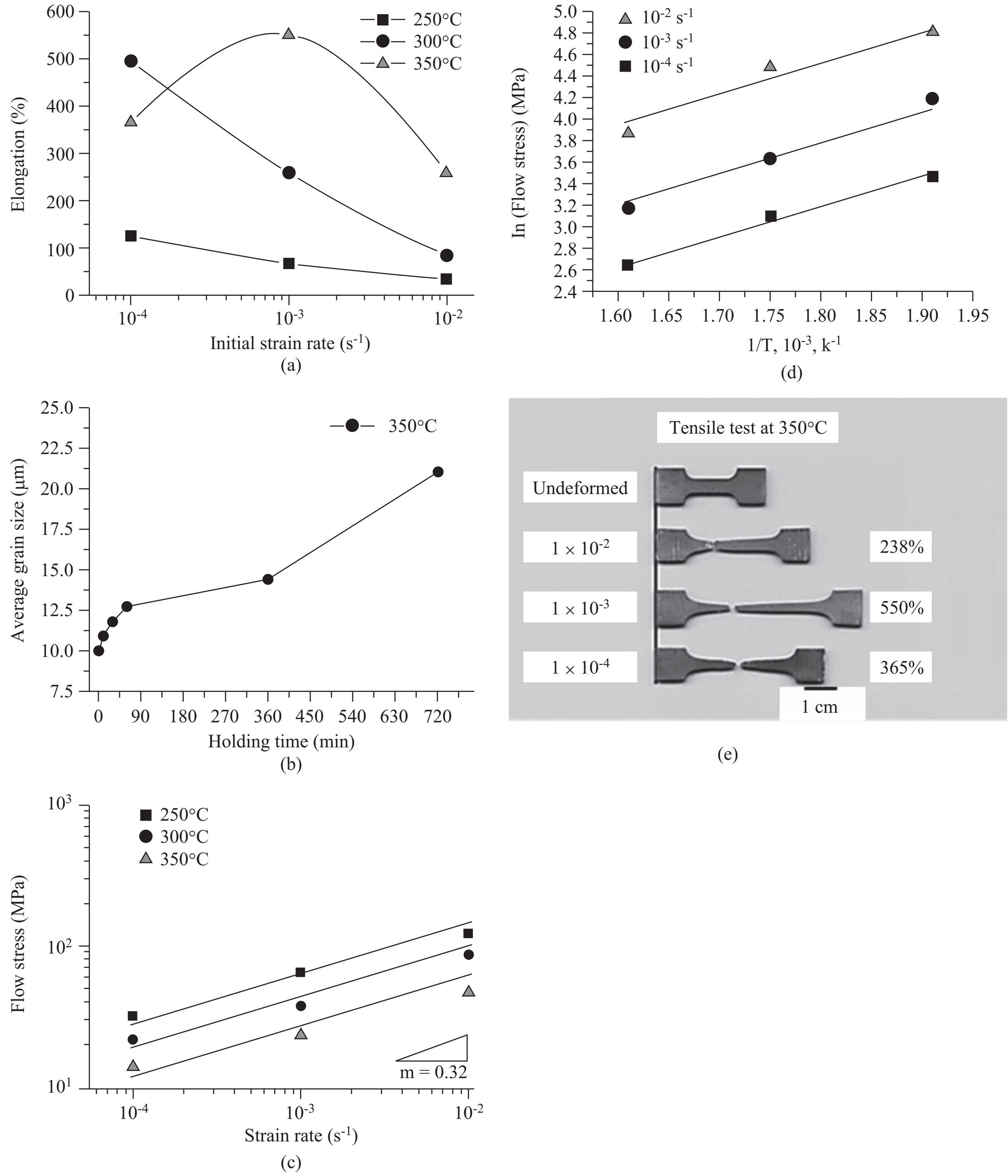

Fig. 2. **(a) Elongations obtained at different strain rates and temperatures, (b) Average grain size after heat treatment at 350°C with different holding time, (c) The m value of different temperatures, (d) Activation energy curves of ln(flow stress) vs. 1/T, and (e) Broken specimens after tensile test at 350°C with different strain rates.**

time 360 min. However, the average grain size obviously grows at 350°C with holding time 720 min; the average grain size increase from 10 μm to 21 μm. This result provides the evidence to explain that the high temperature 350°C producing more thermal energy to active grain coarsening; then grain coarsening decreases the elongation of Mg-5(mass%) Sn alloy at 350°C with strain rate 1×10^{-4} s^{-1}.

In order to understand the mechanism during superplastic process, the activation energy (Q) is calculated at constant strain rate by using the equation (Wu and Liu, 2002):

$$Q = nR\,[\partial\,(\ln\sigma) / \partial\,(1/T)] \tag{1}$$

where σ is the flow stress; n is the stress exponent (n = 1/m); R and T are the gas constant and absolute temperature. The mean strain rate sensitivity (m value) of 0.32 is obtained from the slope of the curve in Fig 2(c); then ∂ (ln σ) / ∂ (1/T) is estimated from the slope of the curve in Fig. 2(d), thus the activation energy is determined to be 98.7 kJ mol^{-1}.

Noteworthy, Park et al. (2011) investigated that the m value at low strain rate range of Mg-8Sn-1Al-1Zn alloy was 0.37; then the superplastic deformation mechanism of this alloy was controlled by grain boundary sliding (GBS) and grain matrix slip deformation (GMD) (Park and You, 2011). The m value (0.32) of this study is so close to the m value (0.37) of Park et al. (2011) reporting, that suggesting the mechanism of superplasticity in this study is mainly controlled by the GBS + GMD at 250°C, 300°C.

By contrast, the major deformation mechanism at 350°C may transfer gradually from GBS + GMD (1×10^{-2} s^{-1} and 1×10^{-3} s^{-1}) to GMD (1×10^{-4} s^{-1}). Fig. 2(b) shows that the average grain size grows obviously from 10 μm to 21 μm at high temperature 350°C with holding time 720 min. It was well known that grain refinement and resisting grain growth was beneficial for high temperature plasticity which was due to the activation of GBS; however, grains coarsening are performed by high temperatures or low strain rates (Sherby and Wadsworth, 1989).

Furthermore, the deformation mechanism of GBS and GMD competed against each other at high temperatures (Park and You, 2011).

Therefore, grain coarsening can reduce the opportunity of GBS; thus, the GMD become a key role to control the deformation mechanism during tensile test at high temperature 350°C with the lowest strain rate 1×10^{-4} s^{-1}.

Moreover, we calculate the active energy (Q) of 98.7 kJ mol^{-1} in the present study. This value is higher than grain boundary diffusion (95 kJ mol^{-1}) but much lower than lattice self-diffusion (135 kJ mol^{-1}) of magnesium alloy (Frost, 1982; Somekawa, 2005). It means that the grain boundary diffusion may dominate the superplastic deformation mechanism during tensile test at high temperatures.

The undeformed and broken specimens are shown in Fig. 2(e). The maximum elongation reaches 550% at high temperature 350°C with strain rate 1×10^{-3} s^{-1}. The necking phenomenon which was very similar to those earlier reports for a Mg-Based alloy (Matsubara, 2003; Lin, 2005) was occurred when the strain rates were 1×10^{-2} s^{-1} to 1×10^{-4} s^{-1}.

3. Fracture Surface

The failure of magnesium alloy was usually brittle through cleavage with a H.C.P structure (Yizhen et al., 2000). Fig. 3 shows SEM image of the tensile fracture surface. Fig. 3(a) is the fracture surface of specimen after ECAE six passes which conduct tensile test with strain rate 1×10^{-2} s^{-1} at 250°C. The result shows that the fracture surface is composed of cleavage. Fig. 3(b) shows that the specimen is broken at 300°C. The result shows that the fracture surface consists dimples and cleavages. It means that the deformation of Mg-5(mass%) Sn alloy was transformed gradually from the brittle to the plastic through thermal activation. Further, when the tensile test temperature increase to the 350°C, the fracture pattern change to the dimples as shown in Fig. 3(c). Compared with Fig. 3(a),

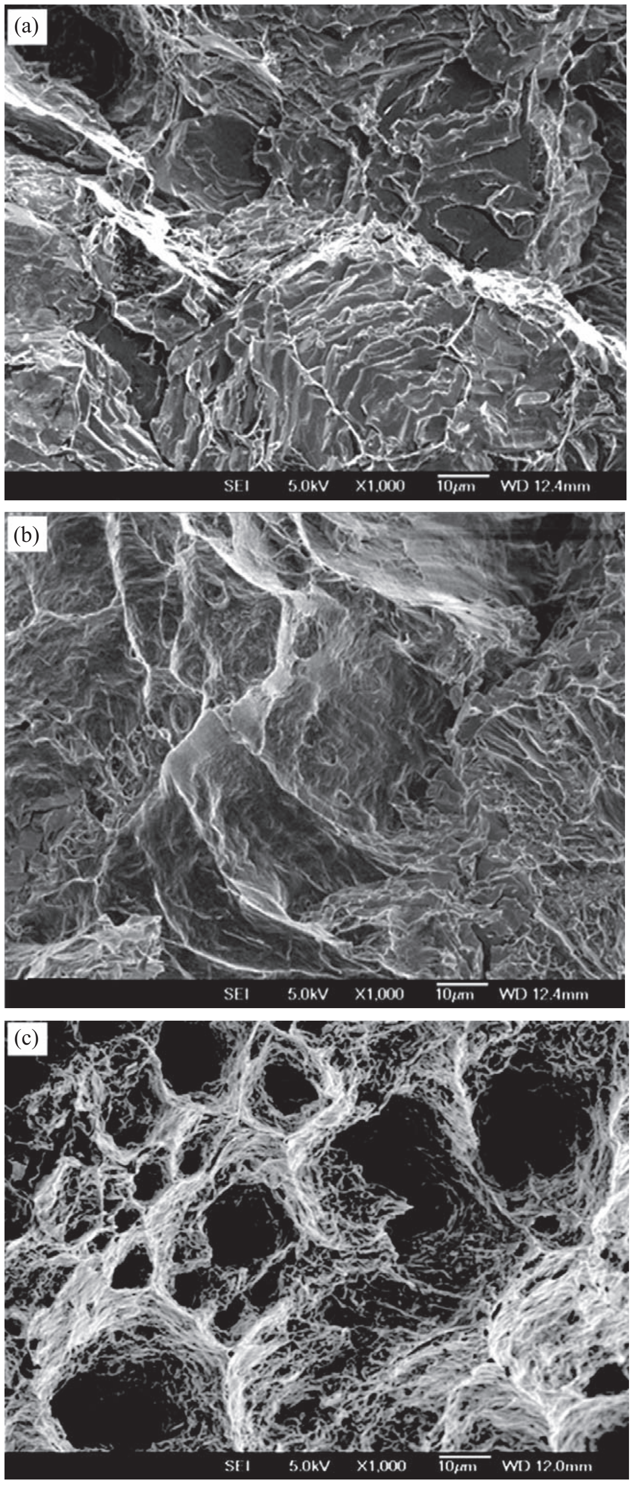

Fig. 3. SEM image of fracture surface after tensile test with strain rate 1×10^{-2} s^{-1} at different temperatures (a) 250°C, (b) 300°C, and (c) 350°C.

all the cleavages disappear very obviously and the major patterns are replaced by dimples.

V. CONCLUSIONS

The grain size of Mg-5(mass%) Sn alloy was reduced from 147 μm (as-cast) to about 10 μm by ECAE six passes. The microstructure of Mg-5(mass%) Sn alloy after ECAE six passes composed of homogeneous fine grains and Mg_2Sn precipitates.

There was no obvious grain growth at 350°C after heat treatment with holding time from 10 min to 360 min. However, the grain growth could be found at 350°C with 720 min, the average grain size increases from 10 μm to 21 μm.

The maximum elongation was conducted at 350°C with a strain rate of 1×10^{-3} s^{-1}; the elongation could reach 550 %.

The superplastic behavior of Mg-5(mass%) Sn alloy was mainly controlled by GBS + GMD. However, the deformation mechanism at 350°C with the lowest strain rate 1×10^{-4} s^{-1} could change to the GMD. The activation energy (Q) was calculated to be 98.7 kJ mol^{-1}. The grain boundary diffusion dominated the superplastic deformation mechanism during tensile test. The fracture surface was cleavage at 250°C with strain rate of 1×10^{-2} $s^{-1}$and the fracture surface replaced by the dimples was transformed gradually from the brittleness to the plasticity at 350°C.

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