Hot tensile deformation and fracture behavior of ultrafine-grained AZ31 magnesium alloy processed by severe plastic deformation

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Abstract

Parallel tubular channel angular pressing (PTCAP) process as a novel intense plastic deformation method was applied to an AZ31 magnesium alloy tubes to achieve an ultrafine grained (UFG) structure. Tensile tests at different temperatures of 25°C, 350 °C, 400 °C and 450 °C were done on UFG AZ31 samples processed by multipass PTCAP process. The results showed that the second passes PTCAP processed specimen exhibits the best elongation of about 263 % at a temperature of 400 °C. Fractured tensile samples were metallurgically characterized to analyze the fracture mechanism. The result indicated a decreasing trend in the average grain size along the gripper section to failure tip of the fractured UFG sample. However, the hardness increases from gripper region to the end of gauge region near the fracture tip. However, at the fracture tip, hardness collapsed because the cavity and microvoids formed in this area during tensile deformation. Fractographic SEM images illustrated the mix ductile and brittle fracture modes at tensile temperatures of 350 °C and 450 °C. Though, completely ductile fracture mode with distinguishable dimples was seen at the UFG sample fractured at 400 °C.

Keywords: Ultrafine grained; AZ31 magnesium alloy; SPD; PTCAP; Hot deformation; Fracture.

1. Introduction

Among the different ways to reduce fuel consumption and exhaust emission of vehicles, reduction of mass is one of the most powerful and least costly methods. Magnesium and its alloys are the relatively lightest structural metals primarily used as structural alloys due to its light weight. They exhibit relatively high specific strength, good corrosion resistance, and machinability. Different structural applications of magnesium alloys are engine parts, aircraft fuselages, wheels, and digital device cases. They are also used in rockets, missiles, jet-engine parts, portable power tools, and luggage frames. Nevertheless, poor formability and low ductility of these alloys limit their usage [1]. As a result, many attempts have been performed to resolve these restrictions. Severe plastic deformation (SPD) capable of producing ultrafine grained (UFG) and nanograined metals is the main tool for the researcher to improve the formability of magnesium alloy [2, 3]. UFG and nanograined metals show high strengths at room temperature and superplastic behavior at higher temperatures. Equal channel angular pressing (ECAP) [4-6], high-pressure torsion (HPT) [7-9] and accumulative roll bonding (ARB) [10-12] are the most famous of the SPD processes. Kang et al. applied ECAP process on a magnesium alloy AZ31 to investigate the effect of the process on its microstructure and mechanical properties [13]. They reported that the elongation to failure was increased with decreasing the grain size by ECAP processing at increased temperatures. Miyahara et al. studied the superplastic behavior of AZ61 magnesium alloy processed by a combination of extrusion and ECAP. They found that the alloy exhibits exceptional superplastic properties with a maximum elongation of 1320% after

processing by four passes of the combined process [14]. According to the broad industrial application of tubular components, new SPD methods are demanded to fabricate UFG and nanograined tubular specimens. Hence, Faraji et al. [15, 16] presented parallel tubular channel angular pressing (PTCAP) process in which a cylindrical tube is deformed to an extremely large strain without a change in its dimensions. As illustrated in Fig. 1(a), at the first half cycle of the PTCAP process, the tube constrained between the mandrel and die is extruded through the symmetric shear zones so that the diameter of the tube increased to a maximum value. At the second half cycle, the tube is returned to the initial dimension by extruding back to the same shear regions by the second hollow punch as shown in Fig. 1(b). The parameters of PTCAP process were also represented in Fig. 1. Different aspects of UFG tubes fabricated via PTCAP processes such as mechanical anisotropy [17], transmission electron microscopy analyzes and determination of dislocation density [18], and energy absorption capacity [19] were investigated by the researchers. Moreover, there are a few published data on the tubular SPD-processed magnesium alloys [20], and there is almost no published paper about the microstructure evolution of hot deformation and superplastic behavior of AZ31 tubes processed by PTCAP. In this study, hot deformation behavior of AZ31 magnesium alloy tubes processed via multipass PTCAP is investigated. Also, the influence of tensile test temperature on the microstructural evolution during the tensile test is examined. Moreover, the fracture mechanism of UFG AZ31 alloy tube during tensile loading at elevated temperatures is investigated.

Fig. 1. Schematic of (a) the first half pass and (b) the second half pass of the PTCAP process along with the die parameters [21].

2. Materials and Methods

The experiments were conducted on an as-cast AZ31 magnesium alloy with a chemical composition shown in Table 1. Samples in a tube form with an outer diameter of 20 mm, a thickness of 2.5 mm and length of 40 mm were prepared from the as-cast ingot by machining. A PTCAP die was designed and manufactured from hot work steel (H13) and hardened up to 50 HRC. The PTCAP tests were performed through one, two and three passes by an INSTRON

press machine at a temperature of 300 °C at a ram speed of 10 mm/min. Molybdenum disulfide (MoS₂) was applied as the lubricant to reduce the friction between contacting surface of dies and sample [21]. Mechanical properties and microstructural evolution of unprocessed and PTCAP processed tubes were characterized. The tensile test specimens with 4 mm in gage length lying parallel to the longitudinal axes of the tube with the cross-sectional area of 2.5×3 mm² were prepared using wire electro-discharge machining. First, the samples were tensile tested at a constant temperature 400 °C at a strain rate of 10⁻³ s⁻¹ using an SANTAM tensile testing machine. Then, the tensile sample prepared from two passes PTCAP processed tube were tensile tested at different temperatures of 350 °C, 400 °C, and 450 °C. Three separate samples were tested for each experimental condition to warrant good reproducibility. Subsequently, the microstructures of tensile samples after the test were characterized using optical microscopy (OM) along the tensile loading. The samples were mechanically polished and then etched for 5 s using a solution of 4 g picric acid, 10 ml acetic acid, 70 ml ethanol, and 20 ml distilled water. The fractured surface morphologies were observed by scanning electron microscopy (SEM) using a Hitachi-FESEM. Vickers microhardness tests were accomplished using COOPA model microhardness machine at a load of 200 g applied for 10 s.

Table 1. The chemical composition of AZ31 magnesium alloy

Element	Al	Zn	Mn	Si	Fe	Cu	Mg
Mass (%)	2.90	0.95	0.20	0.013	0.15	0.01	Bal.

3. Results

Fig. 2 shows typical OM microstructure of as-received and PTCAP processed tubes after first, second and third passes. The homogeneous microstructure of the as-received material with a mean grain size of $\sim 520 \mu\text{m}$ is observed in Fig. 2(a). Figs. 2(b-d) illustrate the microstructure of multipass PTCAP processed tubes. As shown, new finer grains formed along the initial grain boundaries. The necklace-like arrangement named bimodal structure in Figs. 2(b-d) was also observed in the previous studies [22, 23].

The influence of the number of PTCAP passes on the stress-strain curve of the AZ31 samples at an initial strain rate of 10^{-3} s^{-1} at a testing temperature of 400°C is shown in Fig. 3. Inspection shows that the flow stress and ductility of the first and second pass PTCAP processed samples are higher than that of the as-received sample. Though, lower strength is seen for the third pass PTCAP processed sample. It is clear that the yield strength of the third pass processed sample decreases to $\sim 15 \text{ MPa}$ from an initial value of $\sim 45 \text{ MPa}$. As shown in Fig. 3, the second pass PTCAP processed sample exhibits 263 % elongation to failure at 400°C . Therefore, to investigate the influence of temperature, second pass processed sample was selected to pull out at various temperatures of 350°C , 400°C , and 450°C .

Fig. 2. OM microstructure of as received (a), and PTCAP processed tube through first (b), second (c), and third (d) passes.

Fig. 3. Engineering stress-strain curves of as received and multipass PTCAP processed samples tested at 400 °C at an initial strain rate of 0.001 s^{-1} .

Fig. 4 shows engineering stress-strain curves of the second pass PTCAP processed sample tested at various temperatures at an initial strain rate of 0.001 s^{-1} . As shown, the flow stress increases rapidly with increasing strain to reach a maximum value at the certain strain. Then, the flow stress continuously decreased in all curves. At the initial stage of the tensile test, the strain hardening occurred due to the accumulation of dislocations [24]. After peak stress, the softening behavior occurred due to the initiation of dynamic recrystallization and grain growth at elevated temperatures [25]. The highest strength is found at a temperature of $350 \text{ }^{\circ}\text{C}$, though the sample was rapidly fractured at a relatively low elongation of $\sim 79 \%$. In contrast, at $400 \text{ }^{\circ}\text{C}$, the sample reveals a more active dynamic process and the elongation reached to 263% . Though, $\sim 50 \%$ lower peak strength is seen in comparison with $350 \text{ }^{\circ}\text{C}$ counterpart.

Fig. 4. Engineering stress-strain curves of second pass PTCAP processed at different temperatures at a strain rate of 0.001 s^{-1} .

Figs. 5-7 show OM micrographs of the fractured tensile test of the second pass PTCAP processed samples at different regions (near the fracture tip, gripper region, and gauge regions)

tested at a strain rate of 10^{-3} s^{-1} at 350 °C, 400 °C and 450 °C, respectively. At elevated temperatures, the non-basal slip systems like pyramidal and prismatic slip become active. This leads to improvement in the ductility [26]. Nevertheless, some undesirable phenomena such as strain concentration and cavity formation can aggravate the ductility [27, 28]. Fig. 5 illustrates the OM microstructure of different positions of the tensile sample tested at 350 °C. As it reveals, the bimodal structure remains with few rounded cavities in the fractured tip region. Fig. 6 exhibits the microstructures of the tensile specimen tested at 400 °C. Fracture tip at 400 °C shows more necking with a wider range of elongated cavities size. The spread of cavity formation depends on three factors of nucleation, growth and inter-linkage [29-31].

Fig. 5. OM microstructure of the various positions of the fractured tensile test sample (prepared from two passes PTCAped sample) deformed at 350 °C.

Fig. 6. The microstructure of various positions of the fractured tensile test sample (prepared from two passes PTCAPed sample) deformed at 400 °C.

At a temperature of 450 °C, as shown in Fig. 7, the recrystallized grains grow up and leads to a decrease of the strength. It also caused the average grains size to become 14 μm. It is widely known that DRX takes place by grain boundary diffusion and migration process [25]. The increase of the deformation temperature can strengthen the grain boundary diffusion and migration process which assist to activate the grain boundary sliding as a dominant deformation mechanism. The distinct bimodal structure was observed at a temperature of 350 °C in the gauge region, however in 400 °C sample, few fine grains was evident besides coarse grains. Though, almost equiaxed grains appear in the sample tested at 450 °C due to DRX. The microstructure of

gripper sections is coarser than other positions in all samples due to static annealing and no straining.

Fig. 8 represents that the overall area of fracture tip region covered by cavities are 3.5%, 14%, and 5% at various test temperatures of 350 °C, 400 °C, and 450 °C, respectively. It means that the density of cavities at 350 °C is smaller than others. Also, at 400 °C, the density of cavities is largest, and the cavity shapes are almost irregular.

Fig. 7. The microstructure of the different positions of the fractured tensile test sample (prepared from two passes PTCAped sample) deformed at 450 °C.

Fig. 8. The fraction of cavities at the fracture tip versus test temperature.

Fig. 9(a) shows the one-half of the fractured tensile sample (prepared from two passes PTCAPed sample) with tracking points (*a* to *i*) for evaluating the distribution of microstructure and mechanical properties. Fig. 9(b) represents the distribution of the grain size of the tracking points (points *a* to *i*) at various test temperatures. From this figure, three distinguished results can be explained. First, a decreasing trend of grain size from positions *a* to *i* is evident in all samples. At position *i*, the enormous strain was applied, and dislocation accumulation besides DRX causes the formation of new finer grains. Second, the minimum average grain size belongs to the fracture tip region of the sample tested at 400 °C. It is because of more imposing strain applied at this region during the tensile deformation and necking stage. Third, the different slopes of the curves were seen. It can be observed that required energy for grain growth is not supplied at 350 °C. In contrast, at 450 °C, the striking discrepancy appears between gripper region and the fracture tip. In the other word, noticeable grain growth takes place at 450 °C results from static annealing.

(a)

(b)

Fig. 9. (a) One-half of the fractured tensile sample (prepared from two passes PTCAPed sample) with tracking points (points *a* to *i*) and (b) the distribution of the grain size in the tracking points (points *a* to *i*) at various test temperatures.

Fig. 10 shows the variation of hardness along the one-half of the fractured tensile samples along points *a* to *i* (as shown in Fig. 9(a)) at various temperatures. As the temperature increases, the hardness value decreases. At 450 °C, grains growth is dominant, and consequently, hardness reduces in comparison to 350 °C and 400 °C cases. Clearly, in the gripper region, hardness remains almost unchanged but in the gauge length region, the hardness increases rapidly due to applying strain and more grain refinement. Meanwhile, the hardness in the fracture tip falls in all the curves of Fig. 10.

Fig. 10. Variations of hardness along the fractured tensile test sample.

Figs. 11 (a-c) shows SEM fractographic images of fractured surface of the tensile specimens (prepared from two passes PTCAPed sample) under the strain rate of 10^{-3}s^{-1} at various temperatures of 350 °C, 400 °C, and 450 °C. In general, localized cone-shaped necking region and macroscopic fracture with many microvoids can be found [26]. Fig. 11(a) shows that localized smooth neck with small dimples in the diversity of size and depth is seen. This indicates a combination of ductile and brittle fracture modes. Fig. 11(b) exhibits localized cone-

shaped necking. The fractured surface consists of many small dimples and sharp tearing edge that indicates a specific characteristic of a ductile fracture mode. Moreover, at elevated temperatures, deformation mechanism of grain boundary sliding is activated. Therefore, the elongation to failure at 400 °C is ~200 % which is greater than that at 350 °C. From Fig. 11(c) a sharp localized necking with high reducing rate in the cross section could be seen.

4. Discussion

The bimodal microstructure of Figs. 2(b-d) appears when the unprocessed grain size is greater than a critical value [24]. Although further studies are now required for accurate quantifying of critical value, it depends on the alloy composition and processing temperature and also on the presence of a back-pressure or compressive hydrostatic pressure of the SPD method [25]. Grain boundaries as high energy regions are suitable for nucleation of new recrystallized grains as a result of the development of stress concentration and subsequent activation of basal and non-basal slip systems. Applying the further deformation up to third PTCAP pass (Fig. 2(d)) leads to a mix of equiaxed coarse and finer grains. This indicates a dynamic balance between the grain refinement by strain and grain growth occurred during PTCAP process of AZ31 at elevated temperatures. Thus, there exists a critical PTCAP pass number at a given temperature for obtaining a balance microstructure. Same phenomena were also reported in Ref. [32]. This observation confirms that the bimodal grain structure is transitional in nature and may be changed by imposing more strains.

As shown in Fig. 3, a softening behavior could be seen in all strain-stress curves. Softening behavior is more predominant as a consequence of the grain refinement by dynamic recrystallization and grain growth during the hot tensile testing [13]. During hot deformation, it

was observed that the equiaxed shape and relatively small size of the grains remain unchanged after large elongations. Though, an additional strain enhanced the grain growth. It may be attributed to the fact that the grain neighbors change by relative grain translation results from grain boundary sliding [33, 34].

Fig. 11. SEM photomicrographs of the fractured surface of the tensile test sample at different temperatures.

As observed in Fig. 4, the strength is decreased with increasing the test temperature. It is worth noting that the elongation at 400 °C is much greater in comparison with 450 °C. This behavior can be considered as the result of two reasons. First, 450 °C is above the eutectic temperature (~430 °C) [35], and inverse eutectic melting reaction ($\alpha+\beta=L$) take place. It leads to the higher mobility of dislocation during plastic deformation. Second, DRX happened, and grain growth was perceived in the microstructure of the deformed sample. Table 2 compared published data on the high-temperature elongation of AZ31 magnesium alloy with the result of the current study.

Table 2. Summary of publications trying to achieve high ductility in AZ31.

AZ31 Initial condition	Initial GS (μm)	Production method	Final GS(μm)	Strain rate (s^{-1})	Temperature (°C)	Elongation (%)	Ref.
Extruded	5.4	ECAP, 8P (200 °C)	1	1×10^{-3}	200	200	[36]
Extruded	35	HPT, 10 turns , RT, 6 GPa	110 nm	1×10^{-3}	200	282	[37]
As-cast	223	ECAP, 5P (300 °C)	3.8	1×10^{-2}	350	156	[38]
Extruded	9.4	ECAP, 6P (200 °C)	1.6	3.3×10^{-4}	200	210	[25]
As-cast	-	Extrusion (300 °C) +Rolling (200 °C),	500 nm	5.0×10^{-4}	250	227	[39]
As-cast	520	PTCAP (300 °C), 2P	8	3×10^{-3}	350	79	This study
As-cast	520	PTCAP (300 °C), 2P	8	3×10^{-3}	400	263	This study
As-cast	520	PTCAP (300 °C), 2P	8	3×10^{-3}	450	145	This study

It is evident that the elongation to failure depends on the grain size. AZ31 alloy with 110 nm grain size processed by HPT showed an elongation of about 282% [37] while the same alloy with one μm grain size processed by ECAP showed 200% elongation [36]. Also, it could be obviously seen that the temperature of the tensile testing has a significant effect on the elongation.

Plastic deformation during the hot tensile test mostly occurred in the grain growth step, whereas interlinkage is the main reason of the initiation of the failure. As shown in Fig. 6, during diffusional cavity formation mechanism, vacancies cluster together and form cavities via tensile stress after overcoming free-energy barrier [40]. These cavities could be seen in the fracture tip regions of all samples. Near the final fracture, flow stress apparently drops due to the cavitation effect. Before final fracture, the density of cavities and their size becomes larger. This leads to the cavity coalescence and intergranular separation. When the test temperature increased to 400 °C from 350 °C, the grain size reaches to $\sim 6 \mu\text{m}$. In the other word, the new grains easily generate with the increase of the temperature via DRX process at previously formed grain boundaries [41]. It can be concluded from Fig. 8, when the temperature changed from 350 °C to 400 °C, the relative strain contribution at the grain boundaries increases. This assists the opening of voids in the grain boundaries. Nonetheless, at 450 °C, the density of cavities is less than that in the 400 °C. As is believed, increased diffusional accommodation at the higher temperatures is responsible for minimizing the strain concentration at the grain boundaries. Partly, elimination of some pre-existing defects during thermal stabilization can be responsible for this variation of cavities density [42].

The hardness of the fracture tip region falls in all curves of Fig. 10. This trend can be explained by this fact that the microvoids and cavities appeared during hot tensile deformation. Minimum hardness, as expected, was occurred in fracture tip of deformed sample at 400 °C that containing

lots of cavities and microvoids during the period of hot deformation. As shown in Fig. 11, it is evident that at high-temperature deformation, grain boundary sliding is more active than dislocation slip. Therefore, ductility drops due to grain boundary sliding. Rapid grain growth at 450 °C causes to make the brittle fracture mode as dominant fracture mechanism [31]. Finally, the diffusional necking plays an important role in deformation process after yielding [26].

5. Conclusion

UFG AZ31 magnesium alloy tubes fabricated by PTCAP process through first, second and third passes and mechanical and microstructural evaluation during the hot tensile test were investigated at different temperatures. Following items could be concluded from this study:

- The bimodal structure appeared after PTCAP process because the initial grain size of 520 μm is larger than a critical value.
- The grain size was obviously affected by a number of PTCAP passes.
- The hot tensile tests revealed that second pass PTCAP processed sample exhibited 263% elongation because grain boundary sliding is a dominant deformation mechanism at a temperature of 400 °C.
- Nucleation was initiated from grain boundary at a temperature of 400 °C, and a grain growth phenomenon happened at 450 °C.
- Analysis of the microstructure of the fractured tensile sample showed that lots of cavities were observed in fracture tip in all samples, largely at 400 °C temperature.
- The average grain size illustrated decreasing trend from gripper section to fracture tip.

- Hardness dropped at failure tip because of presence considerable amount of cavities and microvoids.
- SEM photos of fractured surface illustrated the combination of ductile and brittle fracture at a temperature of 350 °C.
- The fractured surface at 400 °C was revealed completely ductile fracture mode. However, with increasing the temperature to 450 °C, fracture mode tends to be a mix of ductile and brittle.

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Figure captions

Fig. 1. Schematic of (a) first half pass and (b) second half pass of the PTCAP process with the die parameters [21].

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Fig. 3. Engineering stress-strain curves of as received and multipass PTCAP processed samples tested at 400 °C at an initial strain rate of 0.001 s^{-1} .

Fig. 4. Engineering stress-strain curves of second pass PTCAP processed at different temperatures at a strain rate of 0.001 s^{-1} .

Fig. 5. OM microstructure of the various positions of the fractured tensile test sample (prepared from two passes PTCAPed sample) deformed at 350 °C.

Fig. 6. The microstructure of various positions of the fractured tensile test sample (prepared from two passes PTCAPed sample) deformed at 400 °C.

Fig. 7. The microstructure of the different positions of the fractured tensile test sample (prepared from two passes PTCAPed sample) deformed at 450 °C.

Fig. 8. The fraction of cavities at the fracture tip versus test temperature.

Fig. 9. (a) One-half of the fractured tensile sample (prepared from two passes PTCAPed sample) with tracking points (points *a* to *i*) and (b) the distribution of the grain size in the tracking points (points *a* to *i*) at various test temperatures.

Fig. 10. Variations of hardness along the fractured tensile test sample.

Fig. 11. SEM photomicrographs of the fractured surface of the tensile test sample at different temperatures.

Table captions

Table 1. The chemical composition of AZ31 magnesium.

Table 2. Summary of publications trying to achieve high ductility in AZ31.