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HOT DEFORMATION BEHAVIOR OF Mg-Zn-Al ALLOY TUBE PROCESSED BY SEVERE PLASTIC DEFORMATION

In the current study, severe plastic deformation (SPD) was applied on a commercial Mg-3Al-1Zn alloy tubes via parallel tubular channel angular pressing (PTCAP) route. Different passes of PTCAP process were applied, and microstructure, hardness and tensile properties at the room, and elevated temperatures were evaluated. The results showed that bimodal microstructure appeared and led to AZ31 alloy represented higher hardness, higher strength with a reasonable elongation at room temperature. Similarly, very high elongation to failure was achieved at a higher temperature. The increase in the number of SPD passes up to two, leads to increasing the ductility up to 263% at 400°C. Then, an increase in the number of PTCAP passes to three, leads to decrease in the ductility as the results of formation of microvoids when SPD processing at higher equivalent strains without a sufficient hydrostatic compressive stress. Relatively ductile fracture mode was also occurred in all samples.

Keywords: PTCAP; AZ31 tube; Grain refinement; Hot deformation; High ductility

1. Introduction

Magnesium alloys have recently become more popular because of their recyclability, decent castability, and lowest density between structural metals. So, they are used in numerous automotive parts such as motor parts, dashboards, and steering wheels. However, efforts to increase its strength and broaden their applications have thus far met with limited success because of insufficient ductility as a result of hcp crystal structure [1]. Besides the valuable property of Mg alloys, lower ductility is the most important imperfection not only when processing but also when using as structural parts. It is well-known that grain boundary sliding (GBS) may provide an opportunity for use of the material in high ductility operation even superplastic forming. Clearly, severe plastic deformation (SPD) is common procedure for producing an ultrafine grained (UFG) structure in metallic materials [2]. This motivates the researchers to improve magnesium alloy formability via SPD tool at room and elevated temperatures. Many studies were implemented to investigate the improvement of ductility using grain refinement of Mg alloys for development high tensile ductility at elevated temperatures with the loss of strength [3-7]. Grain boundary sliding in AZ31 magnesium alloy at room temperature up to 523K was investigated in Ref [8]. They stated that above 423K, pure GBS was occurred by resolved applied shear stress acting on grain boundaries. Harai et al. achieved superplastic elongation with a maximum recorded elongation of 620% when testing at a temperature of 473 K in AZ61 sample processed by high pressure torsion (HPT) [9]. Ding et al. presented a model for understanding the grain refinement process of a Mg alloy fabricated by equal-channel angular extrusion (ECAE). They investigated that for the AZ31 alloy, ultrafine-grained alloy exhibits a high strength accompanied by reasonably good tensile ductility [10]. Superplastic flow study in magnesium alloys processed by ECAE was done by Figueiredo et al. [11]. They represented that tensile testing at elevated temperatures shows that the superplastic properties depend on the processing route in which the maximum elongation to failure may occur either in the early stages of processing by ECAE or after processing through a large number of passes. Miyahara et al. indicated exceptional superplasticity in an AZ61 magnesium alloy via the EX-ECAP process. They represented 1320% elongation in four pass processed sample at 473K [12]. Despite the need for high formability magnesium tubes in a broad range of industrial application, efforts have been rarely undertaken to investigate the high ductility behavior of Mg alloy tubes. The present investigation was motivated by this omission. In the present study to evaluate the potential for achieving higher elongations in the AZ31 alloy tube, after processing by parallel tubular channel angular pressing (PTCAP) through a different number of passes, mechanical and microstructural properties were investigated at room and elevated temperatures.

2. Experimental procedure

An as-cast AZ31 magnesium alloy prepared with 20 mm in outer diameter, 2.5 mm in thickness and 40 mm in length as starting material for processing through PTCAP. The schematic

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of PTCAP process and die parameters were illustrated in (Fig. 1). The PTCAP tests were performed in one, two and three passes by an INSTRON press machine at a temperature of 300°C at a ram speed of 10 mm/min. The sample not only were not removed from the die but also were not rotated between consequent passes. To reduce the friction force during the tests, MoS2 was used on the contacting surface [13]. Metallographic analyzes were used to investigate the microstructural evaluation using optical microscopy (OM). Tensile specimens were machined from the tubes as shown in (Fig. 2a) before and after PTCAP process in which the gage length is 4 mm lying parallel to the longitudinal axes and the cross-sectional area is 2.5×3 mm^2. The tensile testing was performed at room and elevated temperature of 400°C using an SANTAM tensile testing machine at a strain rate of 10^{-3} 1/s. Due to curved geometry of the tensile samples, a special clamping device as shown in (Fig. 2b) was designed and used to make the tests easier to improve its suitability for testing at elevated temperatures [14]. The fracture morphology of the fractured tensile samples was observed by field emission scanning electron microscope (FESEM) model Hitachi S4160 at a voltage of 20 KV. All fractured samples were washed with water-based detergent to remove extraneous and foreign dust and contaminations from the fractured surface. Microhardness measurements were conducted after polishing the surface with microhardness instrument equipped with a Vickers indenter under and 200 g load and 10 sec stop time (Fig. 2a). Three hardness measurements was done for each location. The mean value of three measurements was considered as the hardness of that location.

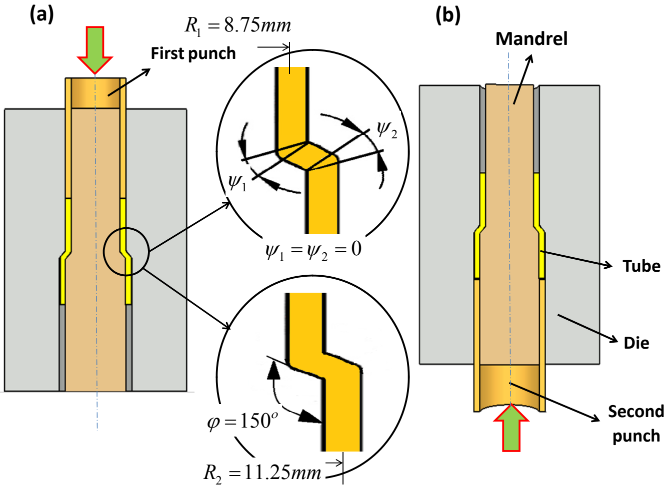


Fig. 1. Schematic of (a) first half pass and (b) second half pass of the PTCAP process along with the die parameters [15]

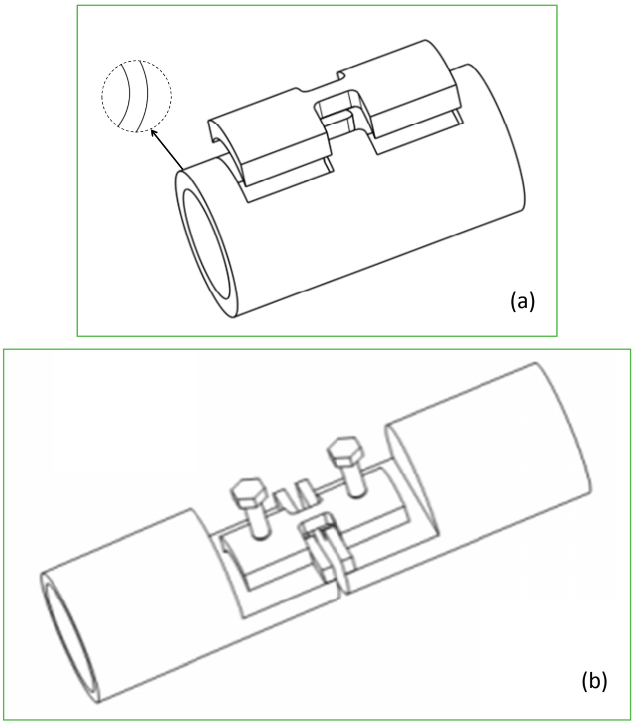


Fig. 2. (a) Tensile test sample, microhardness and OM test positions and (b) clamping device for RT and hot tensile testing

3. Results and discussion

Fig. 3 shows the pictures of unprocessed and PTCAP processed tube at the end of second half cycle. The appearance color after the process is changed because of using MoS2 lubricant at 300°C. (Fig. 4a-d) illustrate the optical microstructures of the unprocessed, first, second and third passes PTCAP processed tubes, respectively. It is clear that a kind of structure with coarse grains surrounded by fine recrystallized grains named bimodal structure that was observed in the previous studies could be seen in PTCAP processed samples [16-18]. Though, after three passes of PTCAP, the microstructure is almost homogenized and also the mean grain size is reduced in comparison with coarse grain counterpart with a grain size of about 520 µm. There is a minimum recrystallized critical grain size (d_c) when processing Mg alloys at temperatures higher than the recrystallization temperature [19]. When SPD processing of Mg alloys at higher temperatures, the dynamic recrystallization occurs and new grains with $\mathrm{d_c}$ in size forms around grain boundaries [19]. The bimodal structure is similar to (Fig. 4b) appeared when the initial grain size is greater than the critical grain size (d_c). Grains were deformed and refined at first PTCAP pass and then less deformed zones and some larger grains were left in the microstructure. The principle of the bimodal structure is that an initial coarse gain where $d > d_c$ when processing by PTCAP leads to the formation of new grains

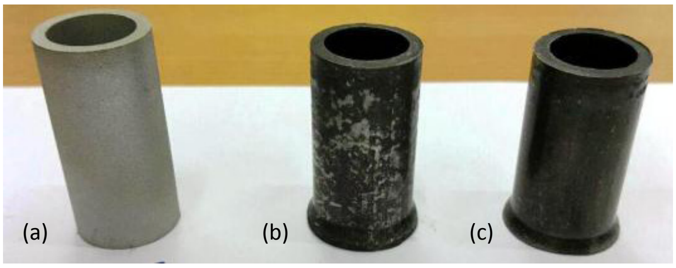


Fig. 3. Pictures of unprocessed (a) and PTCAP processed samples (b) and (c)

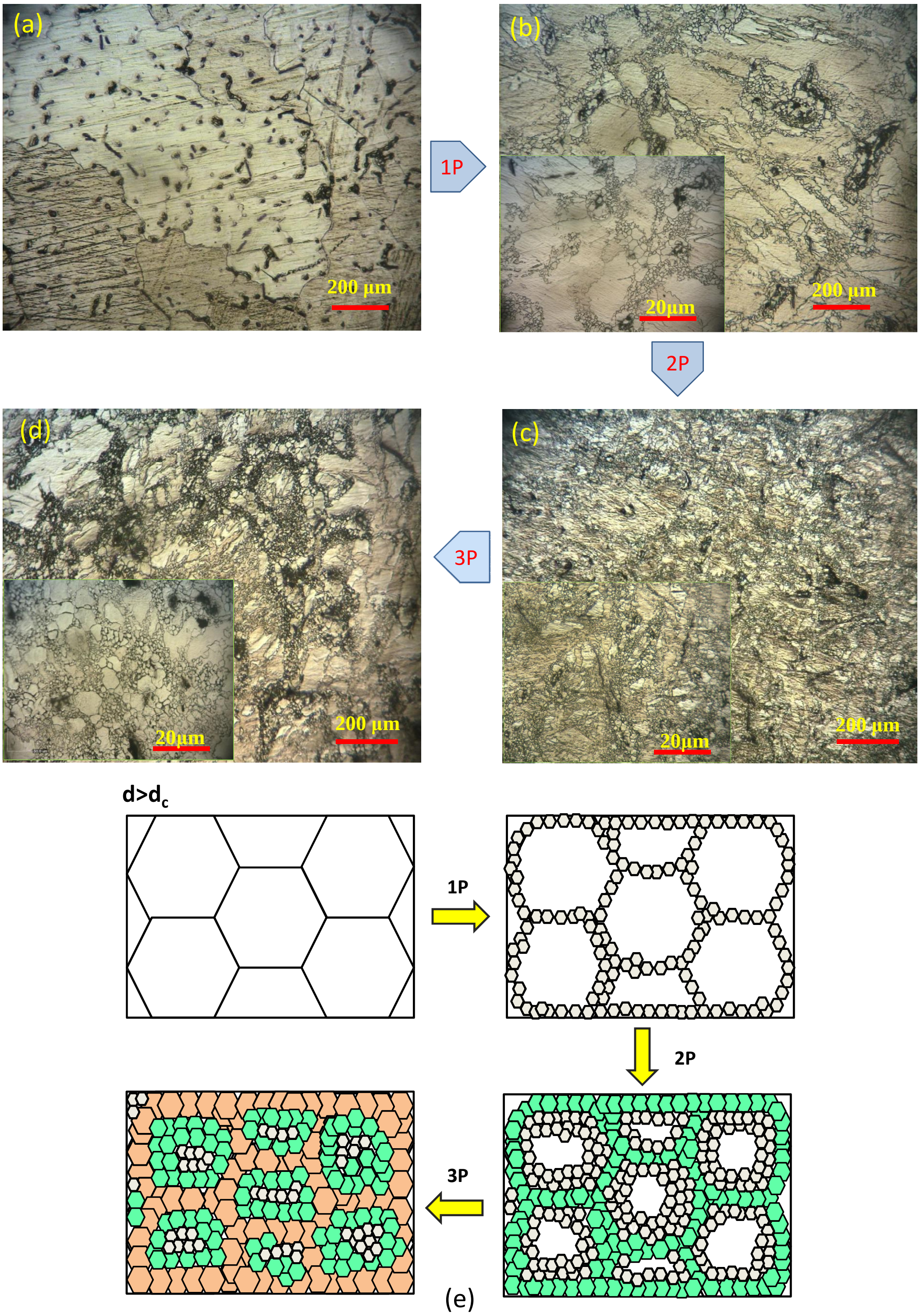
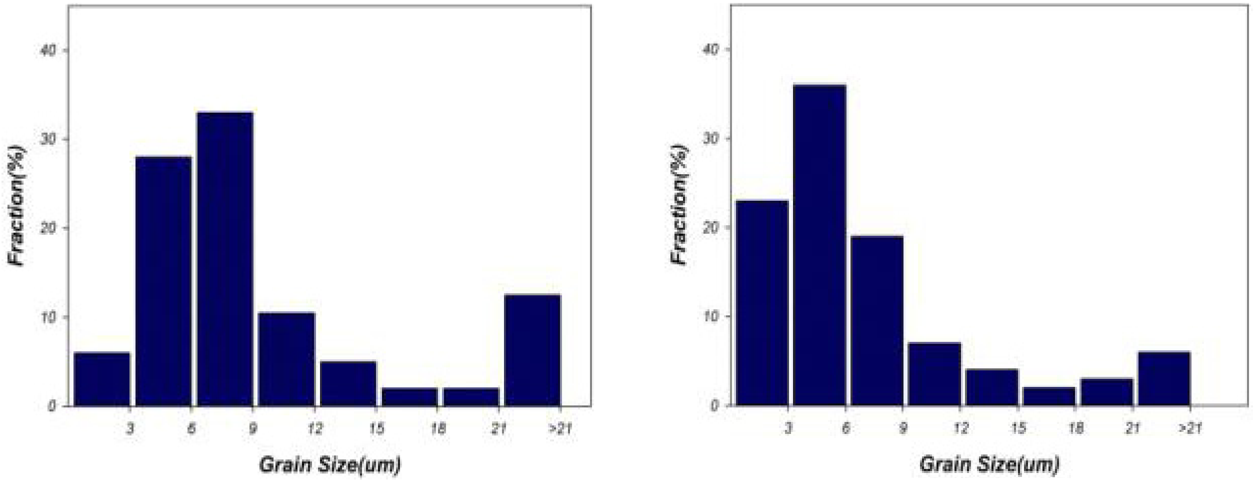


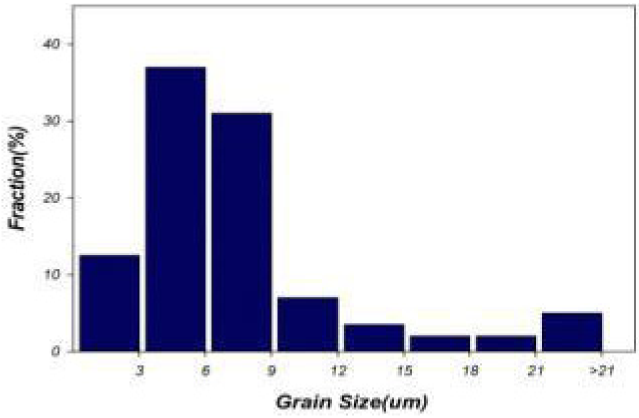
Fig. 4. Optical micrograph of (a) unprocessed, (b) first pass, (c) second pass and (d) third pass PTCAP processed sample, (e) a simplified model for the process of grain refinement of AZ31 during SPD processing (Modified version of [19])

along the initial grain boundaries. However, the initial grain size is now sufficiently large; a bimodal structure is produced wherein the new grains occupy only a fraction of the entire volume of the material, and there are areas in the centers of the larger grains which are not consumed by the formation of the new small recrystallized grains. As PTCAP number of passes increase, more wide UFG regions near the coarse grain boundaries are deformed. Grain refinement in magnesium alloys is therefore characterized by the nucleation of fine grains along the pre-existing grain boundaries. This grain nucleation is related to the stress concentrations at the boundaries and the subsequent activation of both non-basal and basal slip processes [20]. A schematic model for the process of grain refinement of AZ31 during PTCAP processing through a different number of passes was shown in (Fig. 4e). Grain nucleation along the pre-existed boundaries in early PTCAP passes and also their



(a) Mean GS ~11 µm

(b) Mean GS ~8 µm



(c) Mean GS ~9.3 µm

Fig. 5. Grain size distribution of (a) first pass, (b) second pass and (c) third pass PTCAP processed sample

growth by further PTCAP passes leads to an almost equiaxed grain structure in the AZ31 tube. Average grain size and grain size distribution of PTCAPed AZ31 alloy tube after first, second and third passes were respectively shown in (Figs. 5a-c). As depicted, the mean grain size decreased to 11 µm after the first pass due to the accumulated shear strain and dynamic recrystallization during PTCAP. In the second pass, residual coarse grains also divided to finer recrystallized grains with a mean grain size of about 8 µm. After third pass, almost equiaxed grains were observed and the trend of the diagram (Fig. 5c) gets close to normal distribution form. More dynamic recrystallization and also grain growth in the third pass lead to more equiaxed grain structure with a mean grain size slightly larger than that of the second pass processed sample.

Mechanical properties of AZ31 alloy after PTCAP were also investigated using the tensile tests at room temperature and 400°C. The deformation curves of unprocessed and processed specimens for tensile testing at RT and 400°C were plotted as the engineering stress-engineering strain diagrams in (Figs. 6a-b). As shown in (Fig. 5a), it is evident that there is a noticeable increase in the strength after the first pass of PTCAP. The yield strength reached to ~200 MPa from an initial value of ~140 MPa and the tensile strength increased to ~220 MPa from ~160 MPa after one pass. This significant increase strength is the result of grain refinement during SPD processing [21]. There is a subtle point that the elongation to failure did not decrease dramatically after the first pass. It may be attributed to the shape of grains which are equiaxed with high angle grain boundaries. Because the existence of coarse grains surrounded with finer grains, the elongation was not changed drastically. Decrease in strength was took place in the second pass processed sample in comparison with that in the first pass one. In the second pass, regions with small recrystallized equiaxed grains were spread to the wider regions which were not refined in the previous step. Dramatically drop in strength was occurred in the third pass processed sample. Development of microvoids during the further straining is a possible reason for this behavior. AZ31 alloy because of its hcp structure and lack of slip systems, in high imposing strain, macro, and microcrack is appeared and resulting low strength and elongation. Typical plots of engineering stress versus engineering strain for tests conducted on the PTCAP processed tubes at 400°C is represented in (Fig. 6b). As shown a softening behavior could be seen in all curves. Softening effect is more predominant as a consequence of the grain refinement by dynamic recrystallization and grain growth during the tensile testing [22]. The highest elongation of ~263% was achieved in the second pass processed sample. The appearances of the specimen after tensile testing are shown in (Fig. 7).

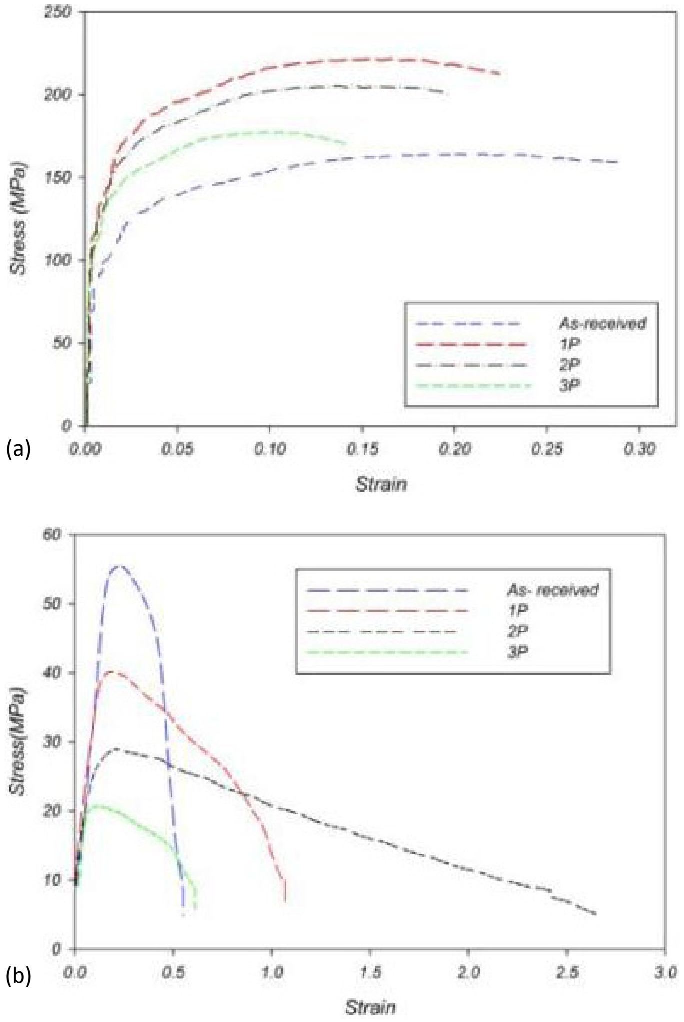


Fig. 6. Engineering stress-strain curves of unprocessed and PTCAP processed samples at (a) RT (b) 400°C

Grain refining promotes grain boundary sliding mechanism, and it is responsible for high ductility behavior in AZ31 material. Tensile elongation versus number of PTCAP processed samples tested at both temperatures of RT and 400°C was exhibited in (Fig. 8). As shown, there is a reasonable trend in the RT test where the elongation is reduced by an increase in the number of passes. However, in the 400°C test, it is revealed that the elongation is reduced after the second pass processed sample. Grain growth and microcrack formation in third pass PTCAP processed sample may be an important reason for this behavior. Table 1 compared published data on elevated temperature elongation of AZ31 with the result of the current study. It may be resulted from the table 1 that the elongation to failure depends on grain size as well established that the superplastic flow mechanism is based on the movement of dislocations along the grain boundaries with some limited intragranular slip acting as an accommodation process [3]. AZ31 alloy with 110 nm grain size processed by HPT showed an elongation of about 310% [23] while the same alloy with 1 μm grain size processed by ECAP showed 200% elongation [6]. Also, comparison of the data showed that the temperature of tensile testing has a remarkable effect on elongation.

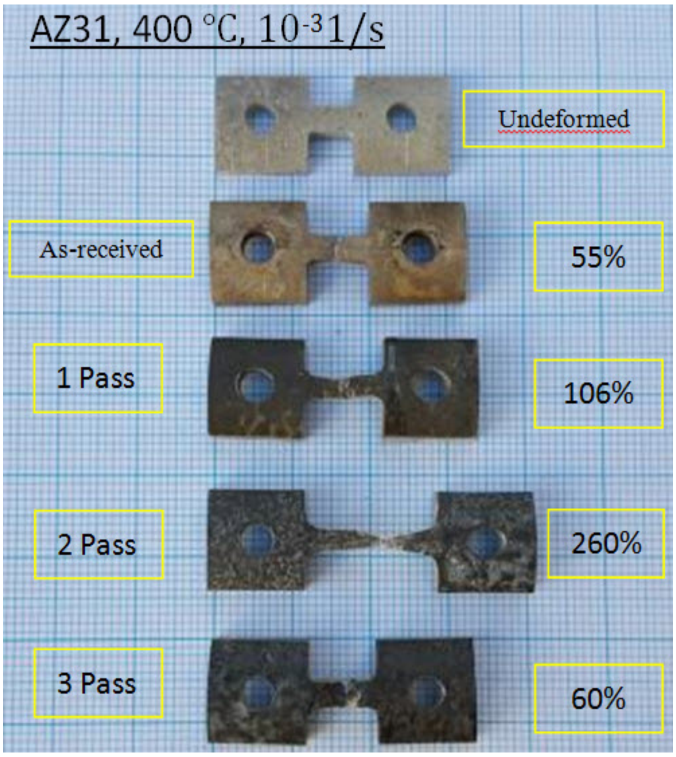


Fig. 7. The appearance of fractured specimens after the tensile test at 400°C

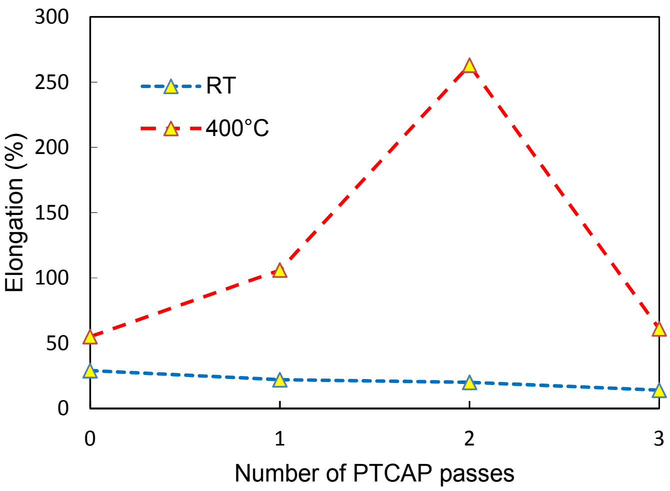


Fig. 8. Elongation versus the number of PTCAP passes diagrams at RT and 400°C tensile testing

Vickers microhardness (Hv) measurements for the unprocessed and PTCAP processed samples have been carried out to evaluate the influence of process and pass number on the hardness behavior of the material. It is found that the first pass PTCAP of AZ31 makes the hardness value to increase suddenly from 38°HV to 61°HV as illustrated in (Fig. 9). Then, hardness value gradually increases at the second pass and reached to 64°HV. However, there is approximately 60% increase in the hardness magnitude of the first pass PTCAPed sample as compared to the unprocessed one. Also, no obvious increase in the hardness value of the second pass PTCAPed sample over the first pass processed one.

TABLE 1

Summary of publications trying to achieve high ductility in AZ31

Process	Grain size (µm)	Temperature of tensile test (°C)	Strain rate in tensile test (s^{-1})	Elongation (%)	Reference
ECAP	1	250	3×10^{-3}	400	[27]
ECAP+ Annealing	15	RT	1×10^{-3}	45	[28]
Rolling+ extrusion+ Annealing	5	400	1.4×10^{-3}	360	[29]
ECAP, 8P, 200°C	3	200	1×10^{-3}	275	[30]
ECAP	No data	400	1×10^{-3}	280	[14]
ECAP, 8P, 200°C	1	200	1×10^{-3}	200	[6]
HPT, 10 turns, RT	0.11	200	1×10^{-3}	310	[23]
ECAP, 4P	3.1	375	1×10^{-3}	169	[5]
PTCAP, 2P, 300°C	8	400	1×10^{-3}	263	Current study

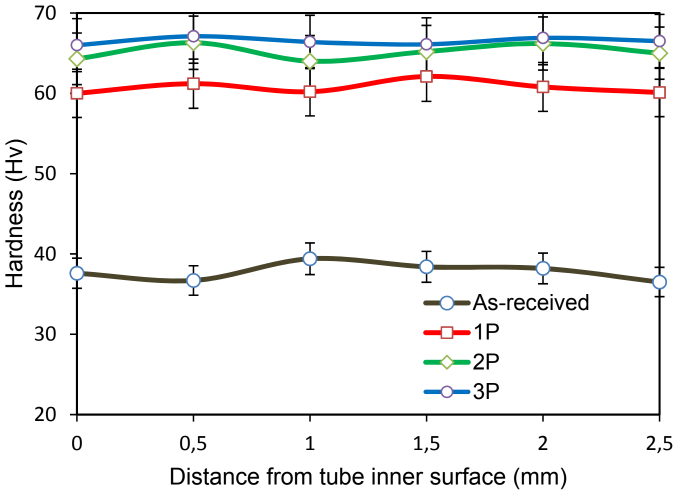


Fig. 9. Microhardness of the unprocessed and PTCAPed tube along the tube thickness

The tensile fracture surfaces analysis was used for interpreting of the fracture mechanism. (Fig. 9) shows the SEM micrograph of fracture surfaces of the tensile specimens that pull out in room temperature and 400°C. It reveals that all the specimens are fractured in a ductile manner, consisting of well-developed dimples over the entire surface. The average dimple size for the initial material is ~10 µm, and it gets reduced to less than 3 µm in the PTCAP processed sample. Drastically decrease in the dimple size may be due to the grain refinement and work hardening, which occurs during severe plastic deformation as reported in the earlier literature [24-26]. As expected, the mechanism of fracture is also affected by the number of the PTCAP passes. In the samples that undergone to further passes of PTCAP process, depth of dimples gradually decreases as illustrated in (Figs. 9b-d) (column 1). It means that samples that undergone higher passes of PTCAP process, due to imposing more strain, experience less deformation before fracture. In the third pass processed specimen that experiences high strain during PTCAP, some micro cracks were appeared as shown in (Fig. 9d). In the hot tensile test (column 2 of Fig. 9), fracture surfaces also experience ductile fracture because of more slip system activated at elevated temperature. The inhomogeneous deformation in adjacent grains is accommodated by slip mechanisms (and even some grain boundary sliding). The appearance of the dimpled fracture surface at 400°C is consistent with intragranular failure due to void formation and coalescence from dislocation activity.

4. Conclusion

AZ31 magnesium alloy were processed via PTCAP, and mechanical and microstructural characteristics were investigated. Following items could be concluded:

- Because of initial grain size is greater than the critical value, the bimodal structure appeared after PTCAP process.
- A model for grain refinement of AZ31 during SPD process was presented.
- The strength of AZ31 alloy at room temperature was increased ~38% after PTCAP with the negligible losing of ductility.
- Two pass processed sample reveals ~263% ductility in 400°C tensile test.
- Microvoid and cracks were appeared in the third pass processed specimen leads to less in strength and elongation in comparison with first and second pass processed samples.
- Hardness increase about 60% from initial samples and in the first pass, hardness almost saturated.
- Analysis of fracture surface of tensile samples illustrated ductile fracture mood in all samples and hot tensile sample, elongated dimples changes to equiaxed dimples.

Acknowledgements

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REFERENCES

[1] B.L. Mordike, T. Ebert, Magnesium: Properties – applications – potential. Materials Science and Engineering: A **302**, 37-45 (2001).

[2] G. Faraji, P. Yavari, S. Aghdamifar, MM.Mashhadi, Mechanical and Microstructural Properties of Ultra-fine Grained AZ91 Magnesium Alloy Tubes Processed via Multi Pass Tubular Channel Angular Pressing (TCAP), Journal of Materials Science & Technology **30**, 134-8 (2014).

[3] R. Figueiredo, T. Langdon, Developing superplasticity in a magnesium AZ31 alloy by ECAP, Journal of Materials Science **43**, 7366-71 (2008).

[4] R. Lapovok, Y. Estrin, M. Popov, S. Rundell, T. Williams, Enhanced superplasticity of magnesium alloy AZ31 obtained through equal-channel angular pressing with back-pressure, Journal of Materials Science **43**, 7372-8 (2008).

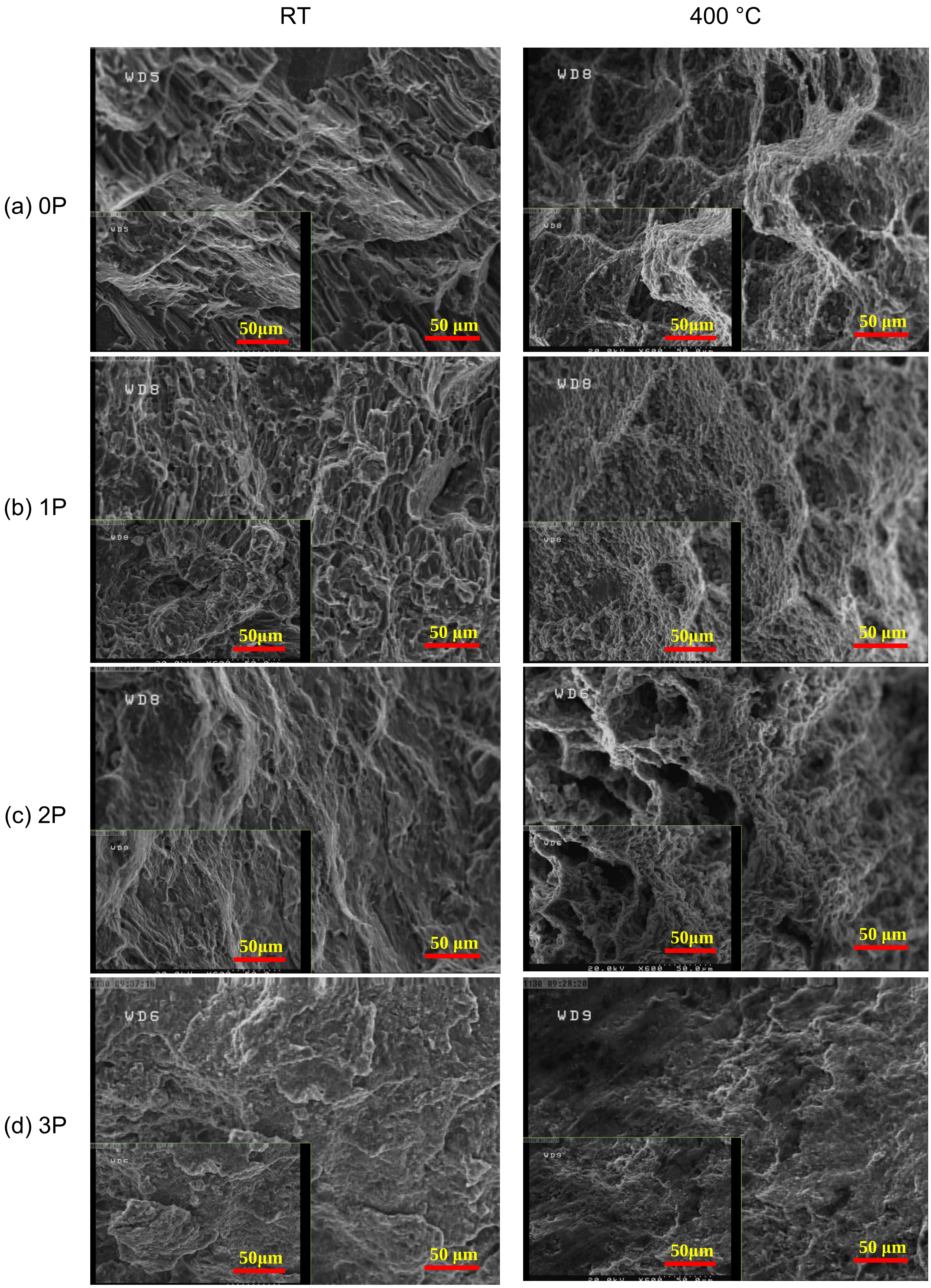

Fig. 10. SEM micrographs of the fractured surface of the tensile test samples at RT and 400°C in (a) as-received, b) first pass, c) second pass and d) third pass PTCAP processed condition

[5] H.G. Svoboda, F.Vago, Superplastic Behavior of AZ31 Processed by ECAP, Procedia Materials Science **9,** 590-8 (2015).

[6] J. Xu, M. Shirooyeh, J. Wongsa-Ngam, D. Shan, B. Guo, T.G. Langdon, Hardness homogeneity and micro-tensile behavior in a magnesium AZ31 alloy processed by equal-channel angular pressing, Materials Science and Engineering A **586**, 108-14 (2013).

[7] R.B. Figueiredo, T.G. Langdon, Evaluating the superplastic flow of a magnesium AZ31 alloy processed by equal-channel angular pressing, Metallurgical and Materials Transactions A **45**, 3197-204 (2014).

[8] J. Koike, R. Ohyama, T. Kobayashi, M. Suzuki, K. Maruyama, Grain-boundary sliding in AZ31 magnesium alloys at room temperature to 523 K, Materials Transactions **44**, 445-51 (2003).

[9] Y. Harai, M. Kai, K. Kaneko, Z. Horita, T.G. Langdon, Microstructural and mechanical characteristics of AZ61 magnesium alloy processed by high-pressure torsion, Materials transactions **49**, 76-83 (2008).

[10] S.X. Ding, C.P. Chang, P.W.Kao, Effects of Processing Parameters on the Grain Refinement of Magnesium Alloy by Equal-Channel Angular Extrusion, Metallurgical and Materials Transactions A **40**, 415-25 (2009).

[11] R.B. Figueiredo, T.G. Langdon, Principles of grain refinement and superplastic flow in magnesium alloys processed by ECAP, Materials Science and Engineering A **501**, 105-14 (2009).

[12] Y. Miyahara, Z. Horita, T.G. Langdon, Exceptional superplasticity in an AZ61 magnesium alloy processed by extrusion and ECAP, Materials Science and Engineering A **420**, 240-4 (2006).

[13] V. Tavakkoli, M. Afrasiab, G. Faraji, M.M. Mashhadi, Severe mechanical anisotropy of high-strength ultrafine grained Cu–Zn tubes processed by parallel tubular channel angular pressing (PTCAP), Materials Science and Engineering A **625**, 50-5 (2015).

[14] F.K. Abu-Farha, M.K. Khraisheh, Analysis of superplastic deformation of AZ31 magnesium alloy. Advanced Engineering Materials **9**, 777 (2007).

[15] M. Afrasiab, G. Faraji, V. Tavakkoli, M. Mashhadi, K. Dehghani, The Effects of the Multi-pass Parallel Tubular Channel Angular Pressing on the Microstructure and Mechanical Properties of the Cu–Zn Tubes, Transactions of the Indian Institute of Metals 1-7 (2015).

[16] K. Bryła, J. Dutkiewicz, P. Malczewski, Grain refinement in AZ31 alloy processed by equal channel angular pressing, Archives of Materials Science **18**, 18 (2009).

[17] G. Vespa, L.W.F. Mackenzie, R. Verma, F. Zarandi, E. Essadiqi, S. Yue, The influence of the as-hot rolled microstructure on the elevated temperature mechanical properties of magnesium AZ31 sheet, Materials Science and Engineering A **487**, 243-50 (2008).

[18] H.K. Kim, W.J. Kim, Microstructural instability and strength of an AZ31 Mg alloy after severe plastic deformation, Materials Science and Engineering A **385**, 300-8 (2004).

[19] R.B. Figueiredo, T.G. Langdon, Principles of grain refinement in magnesium alloys processed by equal-channel angular pressing, J Mater Sci **44**, 4758-62. (2009).

[20] A. Galiyev, R. Kaibyshev, G.Gottstein, Correlation of plastic deformation and dynamic recrystallization in magnesium alloy ZK60, Acta Materialia **49**, 1199-207 (2001).

[21] S. Lee, Y. Saito, T. Sakai, H. Utsunomiya, Microstructures and mechanical properties of 6061 aluminum alloy processed by accumulative roll-bonding, Materials Science and Engineering A **325**, 228-35 (2002).

[22] S.H. Kang, Y.S. Lee, J.H. Lee, Effect of grain refinement of magnesium alloy AZ31 by severe plastic deformation on material characteristics, Journal of Materials Processing Technology **201**, 436-40 (2008).

[23] J. Xu, X. Wang, M. Shirooyeh, G. Xing, D. Shan, B. Guo, et al. Microhardness, microstructure and tensile behavior of an AZ31 magnesium alloy processed by high-pressure torsion, J. Mater. Sci. **50**, 7424-36 (2015).

[24] D.R. Fang, Q.Q. Duan, N.Q. Zhao, J.J. Li, S.D. Wu, Z.F. Zhang, Tensile properties and fracture mechanism of Al–Mg alloy subjected to equal channel angular pressing, Materials Science and Engineering A **459**, 137-44 (2007).

[25] Y.G. Ko, D.H. Shin, K-T.Park, C.S.Lee, An analysis of the strain hardening behavior of ultra-fine grain pure titanium, Scripta Materialia **54**, 1785-9 (2006).

[26] A. Vinogradov, T. Ishida, K. Kitagawa, V. Kopylov, Effect of strain path on structure and mechanical behavior of ultra-fine grain Cu–Cr alloy produced by equal-channel angular pressing, Acta Materialia **53**, 2181-92 (2005).

[27] V. Chuvil'deev, V. Kopylov, M.Y. Gryaznov, A. Sysoev, Low--temperature superplasticity of microcrystalline high-strength magnesium alloys produced by equal-channel angular pressing, Doklady Physics: Springer 343-6 (2003).

[28] T. Mukai, M. Yamanoi H. Watanabe, K. Higashi, Ductility enhancement in AZ31 magnesium alloy by controlling its grain structure, Scripta Materialia **45**, 89-94. (2001).

[29] D. Yin, K. Zhang G. Wang, W. Han, Superplasticity and cavitation in AZ31 Mg alloy at elevated temperatures, Materials Letters **59**, 1714-8 (2005).

[30] H. Lin, J. Huang, T. Langdon, Relationship between texture and low temperature superplasticity in an extruded AZ31 Mg alloy processed by ECAP, Materials Science and Engineering: A. **402**, 250-7 (2005).