Effect of Differential Speed Rolling Temperature into Mechanical Properties of AZ31B Magnesium Alloy

Dr. Azal R. Ismail Production and Metallurgy Engineering Department, University of Technology/Baghdad. Emad A. Hussein Production and Metallurgy Engineering Department, University of Technology/Baghdad. Email:emad_almamoory@yahoo.com

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ABSTRACT

The received AZ31B magnesium alloy sheets of 2 mm in thickness were subjected to differential speed rolling (DSR) process performed on a mill, of which the rotation speed ratio of the lower roll and upper one is kept at constant 1.15, by using the different upper and lower roller diameters. The influence of the rolled sheet temperature on the microstructure of the specimens was examined by optical microscopy, and elongation-to-failure of tensile test at temperature 623 K and initial strain-rate range between $0.5 \times 10^{-3} - 1.83 \times 10^{-3} \text{ s}^{-1}$ was measured. The present process was found to be effective to refine the grain size. Grain refinement became more marked and uniform. The sheet DSRed at 473K exhibited the highest values of 340% and 0.35 for elongation-to-failure and strain rate sensitivity (*m*) respectively.

تأثير درجة حرارة الدرفلةغير المتماثلة على الخواص الميكانيكية لسبيك المغنيسيومAZ31B الخلاصة:

صفيحة مغنيسيوم AZ31B بسمك ٢ ملم خضعت الى عملية در فلة بسرع متفارقة (سرعة در افيل غير متماثلة)، بنسبة سرعة ثابتة مابين الدرفيلين الاسفل الى الاعلى ١.١٥ عن طريق استخدام اقطار مختلفة للدرفيلين. تأثير درجة حرارة الدر فلة على التركيب المجهري للعينات تم فحصه باستخدام المجهر البصري، وتم قياس الاستطالة حتى الفشل لاختبار الشد عند درجة حرارة ٦٢٣ كلفن ومعدلات أنفعال أولية مابين ٥.٥ ⁻¹ - ١٠⁻³ x ^{1.6} ث⁻¹. أظهرت النتائج أن عملية الدر فلة المتفارقة فعالة في تنعيم الحجم البلوري وأصبحت البلورات أكثر أنتظاماً ووضوحاً. أظهرت الصفيحة المدر فلة بدرجة حرارة ٢٧٣ كلفن أعلى قيم لكل من الاستطالة حتى الفشل وحساسية معدل أنفعال (m) حيث

INTRODUCTION

Superplasticity is defined as the ability of materials to afford maximum elongations. In other words, elongation to failure in uniaxial tension when achieve value above 200%. Aluminum and titanium alloys are first used in this field, which exhibit at elevated temperatures superiorly elongations, in last year's focuses of the studies were toward magnesium alloys. Magnesium is widely used in the fields of industry due to high strength to density. The materials with hexagonal close packed structure (hcp) such as Mg alloys exhibit poor formability at room temperature due to the basal slip which has critical resolved shear stress (CRSS) much less than of

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2412-0758/University of Technology-Iraq, Baghdad, Iraq

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non-basal slips. To improvement the formability of Mg alloys many methods were used, one of those is severe plastic deformation in which the alloy subject to high strains [1, 2].

Asymmetrical rolling method, one of the severe plastic deformation (SPD) processes has been extensively used to induce intense plastic strain to the sheet or bulk metallic materials in an attempt to fabricate the ultrafine grained materials which might possess the excellent mechanical properties. Compared with other SPD methods such as high pressure torsion (HPT) and equal channel angular pressing (ECAP), the asymmetrical rolling was allowed to provide the possibility for overcoming the limitation of producing fine grained materials with large dimensions due to its continuous feature in nature. It has been reported that, among the asymmetrical rolling methods, the differential speed rolling (DSR) was considered to be effective for achieving fine grained structure and can introduce shear strain, which can fabricate the different texture compared with the symmetric rolling [3,4].

The objective of this work is to improve the formability of AZ31B magnesium alloy based on fine grain structure by application of superplastic forming (SPF). Investigate further grain refinement through warm rolling of magnesium alloy. The objective is to decrease the average grain size to less than $10\mu m$. Mechanical properties of magnesium alloy are characterized after warm rolling process. The objective is to determine the texture behavior before and after warm rolling.

Experimental work

The as-received material used in this work is AZ31B magnesium wrought alloy sheet with initial thickness 2 mm, and the average grain size was 12 μ m. The chemical composition is shown in Table (1).

| AL | Zn | Mn | Zr | Cu | Si | Fe | Ni | Ca | Sn | Be | Pb | Mg |
|------|-------|-------|---------|---------|--------|---------|---------|---------|---------|---------|---------|-------|
| 2.67 | 0.679 | 0.369 | < 0.001 | < 0.001 | 0.0233 | 0.00292 | < 0.001 | < 0.001 | < 0.005 | 0.00035 | < 0.005 | 96.24 |

Table (1) Chemical composition of AZ31B magnesium wrought alloy sheet

To prepare fine-grained AZ31B magnesium alloy sheet for superplastic deformation, hot rolling tests were performed using a laboratory rolling mill, with small top roll of 47.8 mm diameter. The diameter ratios between the big and small rolls were 1.15. The rotation speeds of upper and lower were 2.6 m/min and 3 m/min respectively. Differential speed rolling was conducted at different temperatures 473, 523 and 573K, these temperatures have been selected within the range of recrystallization temperature for the alloy. Rolling direction was identical to as-received rolling direction. The final thickness was 1.5 mm by one pass (thickness reduction 25%). The samples were pre-heated for 10 minutes before each asymmetric rolling process, also the rolls were pre-heated. Sheets showed good appearance without obvious defects such as edge or surface cracks.

Optical microscopy was utilized to observe microstructure. Specimens for optical microscopy were mechanically polished followed by chemical etching with an acetic picric solution (4.2 g picric acid, 10 ml acetic acid, 90 ml ethanol and 10 ml water) for 5 sec. All optical microstructures were observed along the rolling direction (RD) of the rolled sheets. Tensile test heating system was designed and manufactured from steel as shown in Figure (1). Tensile specimens of dog-bone geometry with the 10 mm gauge length, 5mm width and 2mm shoulder radius were cut along planes coinciding with the rolling direction (RD) Computerized WDW-200EIII machine was used to conduct superplastic tensile tests at temperature 623 K and initial strain-rate range between $0.5 \times 10^{-3} - 1.83 \times 10^{-3}$ S-1 for all DSRed specimens. For the comparison, the testing process was conducted under the same conditions for two specimens as-received and annealed at a temperature of 723K for a period of 7 hours; third specimen was as-received tested at room temperature. The tester was

equipped with heating system was designed and manufactured for this purpose. The true stressstrain curves of the sheets obtained from the tension tests for different strain rates, elongation-tofailure and strain rate sensitivity (m) values were obtained.



Figure (1) Heating system for tensile test specimen

Results and Discussion Microstructure

Figure (2) shows the optical microstructure of the as-received, annealed at 723K for 7 hours and DSRed specimens which are rolled at different temperatures of 473, 523 and 573K of AZ31B magnesium alloy sheets, the annealed structure Figure (2b) shows a fully-recrystallized grain structure, with all grain shapes equiaxed having average size of about (15.7µm). There exists some variation in the grain sizes of the annealed specimen. It is observed that the annealed specimen has larger grain size than the as-received specimen and the DSRed specimens has lower grain size than that of as-received and annealed specimens, due to a more homogeneously distributed and finegrained structure is observed, which suggests that additional shear deformation beneficial for the occurrence of dynamic recrystallization is imposed during DSR process. It is also found from Figures (2c, d and e) that the grain size is increased with the increasing of DSRed process temperature and the lower grain size of about $(3.6 \,\mu\text{m})$ was achieved at temperature of 473K, $(5.4 \,\mu\text{m})$ μm) at 523K and (7 μm) at 573K respectively as shown in Table (2), it is reported by Y. H. Ji [5] that a homogeneous distribution of fine and equiaxed grains of 3µm in diameter was obtained. Weijun Xia [6] the grain size decreases with decreasing rolling temperature in DSR process. A microstructure of differential speed rolling specimens is composed of very small and equiaxed grains which meet the structural requirement of fine grain size less than 10 µm for superplastic deformation. No external cracks observed after DSR process. The microstructural observations show that the DSR process is effective in refining grain size.

| Table | (2) | The | grain | size | of a | s-received, | annealed | and | DSRed | specimens |
|-------|-----|-----|-------|------|------|-------------|----------|-----|-------|-----------|
| | (-) | | 8 | | | | | | | ~ |

| Specimen | Annealed at 723K for 7h | as-received | DSRed 573K | DSRed 523K | DSRed 473K |
|--------------|----------------------------|-------------|---------------|---------------|------------|
| Grain sizeµm | 15.7 | 12 | 7 | 5.4 | 3.6 |



Figure (2) Microstructure of the AZ31B magnesium alloy sheets specimens (a) as-received (b) annealed (c) DSRed 473K (d) DSRed 523K (e) DSRed 573K.

Mechanical properties Elongation-to-failure.

The results of elongation are listed in Table (2), it can be seen from the table that the specimens of DSRed and as-received AZ31B magnesium alloy sheets which tested at temperature of 623K exhibit superplastic deformation at initial strain rate ranging between $0.5 \times 10^{-3} - 1.83 \times 10^{-3} \text{ s}^{-1}$, which indicates that the alloy exhibits notable high strain rate superplasticity. Figure (3) shows the elongation to failure plotted as a function of strain rate for various grain sizes of specimens in this experiment. The elongation of the alloy tends to increase with decreasing strain rate. The DSRed and as-received specimens which were tested at 623 K showed elongation higher than 130% the lowest elongation 135% can be obtained by DSRed 573K at initial strain rate of 1.83×10^{-3} s⁻¹ and the higher elongation of 340% was obtained for the specimen DSRed 473K at initial strain rate $0.5 \times$ 10-3 s⁻¹. The elongation-to-failure increased from 22.3 to 34% with decreasing rolling temperature from 573 to 473 K. In addition Elongation increase in DSRed specimens can be attributed to the grain refinement and microstructural homogeneity. H. Watanabe [7] achieved an increase in elongation from 13.6 to 18.5% with decreasing rolling temperature from 573 to 473 K, and reported that the DSR is suggested to be effective to enhance the room temperature ductility in AZ31 magnesium alloy compared with conventional symmetric rolling. In superplastic deformation, annealed specimen showed improvement in elongation, but it is less than that of DSRed and asreceived within the same testing temperature, because of the annealed specimen has grain size lager and non-homogenous structure, and that not match with the superplastic conditions, compared with the DSRed specimen structure. In addition to that, deformation mechanism of annealed specimen was different from which occurred to the DSRed, and that was cleared in the localized necking, while the DSRed specimen deformation was relatively uniformed and no visible localized necking took place around the fracture, which demonstrated that the deformation mechanism was grain boundary sliding which resulted homogeneous superplastic deformation, as shown in Figure (4). As-received specimen which tested at room temperature showed less elongation due to the limited slip systems, when compared with DSRed specimens, it can be noted that the elongation increased by 96% after the differential speed rolling and superplastic forming processes. In general elongation of AZ31B alloy exhibits significant improve at elevated temperature, as a result of activation of non-basal slip systems.



Figure (3) Elongation to failure as a function of grain size at temperature of 623K for AZ31B magnesium alloy specimens for different initial strain rate.



Figure (4) Tensile test at 623 K for various grain sizes of AZ31B specimens and constant initial strain rates (a) $0.5 \times 10^{-3} \text{ s}^{-1}$ (b) $1.16 \times 10^{-3} \text{ s}^{-1}$ (c) $1.83 \times 10^{-3} \text{ s}^{-1}$.

True Stress-Strain Curve.

Figures (5), (6) and (7a, b) for the DSRed specimens processed at temperatures (473,523 and 573K) Figure (5), the flow stress decreases and strain increases with the decreases in initial strain rate under the same DSRed temperature process, this suggests significant initial strain rate dependencies. Flow stress increases and strain decreases with DSRed temperature process increase under the same initial strain rate conditions Figure (6). The as-received and annealed specimens exhibit the same trend Figure (7a, b). Figure (7c), shows the true stress - strain curves for the as-received AZ31B alloy sheet tested at room temperature at different initial strain rate 0.5×10^{-3} , 1.16×10^{-3} and 1.83×10^{-3} s⁻¹, which exhibited 350 MPa a maximum flow stress value compared with the 9 MPa maximum value of DSRed specimens, which is tested at 623K, due to the lack of sufficient independent slip systems which necessary for deformation of polycrystals. The main slip mode in the magnesium alloys sheets is basal slip. But only two independent slip systems are available in the basal plane, which is not sufficient for homogenous deformation of polycrystals at room temperature.

Figures (8) the curves show that the low initial strain rate lead to increase strain values, where the highest strain stress value of 1.48 at the initial strain rate 0.5 x 10^{-3} s⁻¹ for the DSRed 473K and the lowest value was 0.58 for the specimen annealed at the initial strain rate 1.83 x 10^{-3} s⁻¹. For the DSRed sheet, the flow stresses were much lower than those of the as-received and annealed sheet with the largest decrease of about 17.5 and 30MPa respectively. Further, strains of the DSR processed sheet were improved in comparison with those of the as-received and annealed sheet with the largest increase from 5% to 17%. Weijun Xia [6] suggested that the decrease in flow stress of the DSR specimens may result from the disappearance of twins, while the increase in elongation can be attributed to the grain refinement and microstructural homogeneity. Comparing the asreceived specimen with DSRed processed at 473 and 523 K specimens, the DSRed showed increasing strain value. The DSRed 573 specimen showed a different behavior, where strain values were less, comparing with the as-received specimen because it had a less strain rate sensitivity (*m*value). Annealed specimens were given less strain value by 58% -73% at initial strain rate 1.83 x 10^{-3} s⁻¹ and 0.5 x 10^{-3} s⁻¹ respectively. The results showed that the specimens which had less grain size and high (*m*-value) were given a higher strain.

As the initial strain rate decreasing, the stress-strain curves of the specimens showed a longer steady state flow region and the lower initial strain rate was, a longer time the stable flow stress sustains. It was commonly accepted that the existence of the steady state was the result of a balance between hardening and softening. A long time of stable flow suggested that the progress of necking was extremely gradual in those specimens. It was thought that dynamic recovery or recrystallization occurred and deformation progresses with balancing of work hardening in the steady state flow region.

strain rate = $0.5 \times 10^{-3} \text{ s}^{-1}$

DSRed 523K

1.5

DSRed 473K

1

1

2

2

2

strain rate

1.83x10⁻³ s⁻¹

strain rate

1.16x10-3 s-1

DSRed 523K

1.5

DSRed 473K

DSRed 523K

1.5

DSRed 573K



Figure (5) True stress- strain curves of tensile test at different initial strain rates for AZ31B specimens (a) DSRed 473K (b) DSRed 523K (c) DSRed 573K.



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Figure (7) True stress- strain curves of tensile test at different initial strain rates and different AZ31B specimens (a) annealed test at 623K (b) as-received test at 623K (c) as-received at RT.

Figure (8) True stress- strain curves of AZ31B specimen at different initial strain rate (a) 0.5×10^{-3} s⁻¹ (b) 1.16×10^{-3} s⁻¹ (c) 1.83×10^{-3} s⁻¹.

Figure (9) shows the double logarithmic plot of the relationship between strain rate and flow stress, which was constructed using the tensile elongation tests by reading the flow stress values at a strain of 0.15 in their stress–strain curves. The m-value is a generally recognized assessment basis for the evaluation and comparison of the superplastic potential of a certain material. The higher m-value achieved by grain refinement confirm the great influence of grain boundary sliding on the superplastic behavior. Moreover, it is stated that a fine grain structure moves the optimal temperature elongation range-combination to lower temperatures and higher elongation rates. The (m-value) is defined as:

$$m = \frac{\partial \log \sigma}{\partial \log \dot{\epsilon}} \qquad \dots (2)$$

The values of (*m*) that have been calculated in this study, all of which were greater than 0.2, the maximum value of (*m*) is 0.35 for DSRed 473K with grain size of 3.6 μ m, 0.25 for DSRed 523K with grain size of 5.4 μ m and the minimum value of (*m*) is 0.21 for DSRed 573K with grain size of 7 μ m, which is consistent with obtaining lower elongation in this condition. Y. Q. Cheng reported [8] the enhanced m-value which has been obtained by modification of texture and thus finer microstructure is the reason why a seven times higher fracture elongation could be achieved for AZ91 in tensile testing. Generally, large elongations are obtained where (*m*) values are high [9]. The largest superplastic elongation of 340% has been obtained at initial strain rate of 0.5x10⁻³ s⁻¹ for DSRed 473K specimen, because it had smaller grain size and high (m-value) equal to 0.35 comparing with the other specimens, which were tested at same conditions of tensile test.



Figure (9). The log $\dot{\epsilon}$ vs. log σ curves of the AZ31B alloy

Tensile fracture surface.

Figure (10) show the tensile fracture surface at a temperature of 623K of the DSRed specimens which were elongated up to (340, 325 and 205%) and initial strain rate 0.5 $\times 10^{-3}$, 1.16 $\times 10^{-3}$ and 1.83 $\times 10^{-3}$ s⁻¹ respectively. It is being clearly observed that the specimen is basically fractured intergranularly. The fracture manner is the combination of cleavage or quasi-cleavage between grain boundaries and tearing fracture module. As observed many of refining grains distributed on fracture

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surface with originally integrated shapes, which was obviously the result of grain boundary greatly sliding. There were lots of micro cavities coalescences distributed uniformly on fracture surface, these minute cavities experienced the growth, moving and coalescing in the superplastic deformation [10], lastly resulting in materials discontinuity and final failure. Under this combined mechanism, the DSRed AZ31B magnesium alloy obtained the best superplasticity.



Figure (10). Fracture surface of DSRed 473K after tensile superplastic deformation at temperature of 623K and initial strain rate of (a) $0.5 \times 10^{-3} \text{ s}^{-1}$ (b) $1.16 \times 10^{-3} \text{ s}^{-1}$ (c) $1.83 \times 10^{-3} \text{ s}^{-1}$.

CONCLUSIONS

The conclusions are as follows:

1. The grain size after differential speed rolling was refined for all specimens; uniform microstructures were achieved with less than $10\mu m$.

2. The grain size decreased and strain rate sensitivity exponent (m) increased with lowering the rolling temperature. The extent of fine-grained region was slightly larger in the specimens processed by DSR at 473K than in the DSR at 523K, and then the DSR at 573K.

3. The present alloy exhibited superplastic forming at high strain rate. The maximum superplastic tensile elongation of 340% is obtained at strain rate of 0.5×10^{-3} s⁻¹ of DSRed 473 K, corresponding to a strain rate sensitivity exponent m of 0.35.

REFERENCE

[1].D. Poerschke "The Effects of Forging on the Microstructure and Tensile Properties of Magnesium Alloys AZ31 and ZK60" Case Western Reserve University, Cleveland, OH, USA.

[2].K. Bry, J. Dutkiewicz "Grain refinement in AZ31 alloy processed by equal channel angular pressing" Archives of Materials Science and Engineering, November (2009).

[3].K.U. Kainer "Magnesium – Alloys and Technology" Wiley-Vch Verlag GmbH & Co. KG aA, Weinheim (2003).

[4].Y. He, Q. Pan "Microstructure and mechanical properties of ultrafine grain ZK60 alloy processed by equal channel angular pressing" J. Mater. Sci. 45:1655–1662 (2010).

[5].Y.H. Ji, J.J. Park "Analysis of thermo-mechanical process occurred in magnesium alloy AZ31 sheet during differential speed rolling" Materials Science and Engineering A 485:299–304 (2008).

[6].W. Xia, Z. Chen, D. Chen "Microstructure and mechanical properties of AZ31 magnesium alloy sheets produced by differential speed rolling" J. of Mater. Proce. Tech. 2 0 9:26–31(2 0 0 9).

[7].H. Watanabe, T. Mukai, K. Ishikawa "Effect of temperature of differential speed rolling on room temperature mechanical properties and texture in an AZ31 magnesium alloy" Journal of Materials Processing Technology 182 :644–647 (2007).

[8]. Y. Q. Cheng, Z. H. Chen "Effect of crystal orientation on the ductility in AZ31 Mg alloy sheets produced by equal channel angular rolling" J Mater Sci. 42:3552–3556 (2007).

[9]. U.S. Department of Energy " Innovative forming and fabrication technologies: New opportunities " http://www.osti.gov/bridge.

[10]. Z. Leng , J. Zhang "Superplastic behavior of extruded Mg–9RY–4Zn alloy containing long period stacking ordered phase" Materials Science&EngineeringA576, 202–206 (2013).