Rare Metal Materials and Engineering
Volume 47, Issue 3, March 2018
Online English edition of the Chinese language journal

Available online at www.sciencedirect.com

ScienceDirect

Cite this article as: Rare Metal Materials and Engineering, 2018, 47(3): 0736-0741.

ARTICLE

Microstructure and Mechanical Properties of AZ31B Magnesium Alloy Prepared by Solid State Recycling

Wu Shuyan[1], Ji Zesheng[2], Hu Ming[1], Huang Zhiqiu[1], Tian Chunying[1], Wu Mingzhong[1]

[1] *Jiamusi University, Jiamusi 154007, China;* [2] *Harbin University of Science and Technology, Harbin 150040, China*

Abstract: Machined chips of AZ31B magnesium alloy were consolidated by cold pressing followed by hot extruding at different processing temperatures and extrusion ratios. Compared with ingot extruding alloy, the influences of processing technique on mechanical properties of recycled alloys were analyzed from two main aspects involving the dynamic recrystallization microstructure and the bonding condition between chips. Results show that with the increasing extrusion temperature, the ultimate tensile strength and elongation to failure of the recycled alloys firstly increase and then decrease. The combined action of grain growth and improvement of the bonding between chips with the increasing extrusion temperature results in the variations of mechanical properties of the recycled alloy. With the extrusion ratio increasing from 4:1 to 44:1, the grain is refined, and the bonding between chips is enhanced. This contributes to the increased ultimate tensile strength of recycled alloy. Whereas elongation to failure decreases when extrusion ratio is higher than 25:1 due to the significant deformation strengthening.

Key words: solid-state recycling; AZ31B magnesium alloy; extrusion temperature; extrusion ratio; mechanical property

Having many advantages such as excellent mechanical properties, low density, high dimensional stability and good machinability [1], magnesium alloys show great potential applications in aerospaces, and automobiles[2,3]. However, massive waste chips are produced in the process of machining since magnesium alloys present good machinability. With the increasing application for magnesium alloys, recycling of waste chips attracts more and more attention[4].

For recycling magnesium alloy chips, an advanced recycling method in solid-state has been researched in Japan[5]. Compared with the re-melting process, solid-state recycling shows its advantages of reducing the oxidation loss and energy consumption. The recycled Mg-Al-Ca alloy[6], ZK60[7] alloys and Mg-Zn-Y-Zr[8] alloy processed by the solid-state method exhibit high strength, ductility and superplasticity. The previous researches demonstrated the feasibility of the solid-state recycling. AZ31 alloy is one of the most widely used wrought Mg alloy currently. It is the most suitable to the recycling process in solid state due to the good deformability. However, there was no systemic research for the effects of processing parameters on microstructure and properties of recycled AZ31alloy.

In the present paper, we investigated the variation of microstructure and mechanical property with processing temperature and extrusion ratio of recycled AZ31 alloy and analyzed the reasons.

1 Experiment

Machined chips with the average size of 8 mm×2 mm×0.4 mm were prepared by turning an AZ31B magnesium alloy ingot (Mg-2.9wt%Al-1.0wt% Zn-0.35wt%Mn). The recycled chips were filled into a mold and were cold-pressed into a billet under the pressure of 350 MPa, and the holding time

Received date: March 25, 2017
Foundation item: National Natural Science Foundation of China (51574100, 51404082); Heilongjiang Province Natural Science Foundation (E2015040, E201467); Heilongjiang Educational Office Scientific Research Program (12541793); Jiamusi University Research Project (Lq2012-31); the Opening Foundation of Material Research and Application Key Laboratory of the Harbin University of Science and Technology
Corresponding author: Ji Zesheng, Ph. D., Professor, School of Materials Science and Engineering, Harbin University of Science and Technology, Harbin 150040, P. R. China, Tel: 0086-451-86392500, E-mail: wsy_hit@sina.com

was 1 min. The billet with the mold was heated at a certain temperature for 20 min. Then hot extrusions were conducted in air at an extrusion rate of 0.2 mm/s. With an extrusion ratio of 44:1, samples were extruded at temperatures of 573, 623, 673 and 723 K. Moreover, extrusions were conducted at 673 K with extrusion ratios of 4:1, 11:1, 25:1, 44:1, 100:1. For comparison, extruding was also performed for the AZ31B magnesium alloy ingots under the same conditions and the extruded alloys were called as reference specimens.

Tensile tests were carried out with an initial strain rate of 2.5×10^{-3} s^{-1} on WDW-10 electron universal testing machine, and the tensile axis was parallel to the extruding direction. The tensile test was not conducted for the specimen with extrusion ratio of 100:1 due to its small diameter. Microstructures were obtained through OLYMPUS-GX71-6230A optical microscopy with the observed planes vertical to the extrusion direction. The etching solution contained 2.5 g picric acid, 2.5 mL glacial acetic acid, 5 mL water, and 25 mL alcohol. The average grain size was calculated using the method of linear intercept. The fracture surfaces were investigated by JSM6360-LV scanning electron microscopy.

2 Results and Discussion

2.1 Microstructure and mechanical properties at different extrusion temperatures

Fig.1 shows the microstructures of recycled alloys extruded at different temperatures. After hot extrusion, the boundaries between the chips are not distinguished. The microstructures of all the specimens are composed of small equiaxed grains, and the large twins in the machined chips shown in previous study[9] disappear, indicating that dynamic recrystallization has occurred during the hot extrusion. As shown in Fig.1, when the extrusion temperature rises from 573 K to 673 K, the average grain size increases from 5.5 μm to 7.8 μm. The grain size increases remarkably to 9.8 μm when the extrusion temperature reaches 723 K.

During the extrusion, when the internal energy induced by deformation reaches a certain critical value, the dynamic recrystallization will be activated. The distortion energy difference among grains in deformed metal is the driving force of dynamic recrystallization, and its magnitude is determined by dislocation density and distribution state. Both the number of recrystallization nucleus and its growing rate are related to the deformation temperature in magnesium alloy, that is they are controlled by dislocation motion ability but not by nucleation mechanism. At the lower deformation temperature, it is difficult for dislocations to reunite by moving, so dynamic recrystallization is not easy to occur[10]. Dynamic recrystallization in magnesium alloy is prone to occur due to the limited sliding system, low stacking fault energy and high grain boundary diffusion rate[11], which is accordant with the result that the recycled alloy extruded at lower temperature of 573 K exhibits complete dynamic recrystallization in this study. However, chip-extruded Mg-Zn-Y-Zr alloy contains some non-recrystallized old grains at the processing temperature of 593 K[8]. Dynamic recrystallization should also be related to the composition of the alloy, die construction and ram speed during hot deformation, which implies its complexity. On the other hand, high-migration rate of grain boundaries at high temperature leads to rapid grain growth, so the recycled specimen extruded at 723 K exhibits large grain.

The recycled specimen has slightly smaller average grain than reference specimen at the same extrusion temperature, as stated in the foregoing work[9]. Oxides may bring about additional barriers to the movement of dislocations, resulting in more sites for the piling up of dislocations, which increased the nucleation probability around the precipitates[12]. In the magnesium alloy chips, severe plastic strain and high dislocation density were produced in the machining process which may promote the recrystallization[13]. Furthermore, oxides suppress the grain growth during hot deformation. Therefore, the recycled alloys have finer grains than the reference specimen.

The tensile properties of recycled specimens and reference specimens are shown as a function of the extrusion temperature in Fig.2. For reference specimens, the ultimate tensile strength and elongation decrease with the increasing extrusion temperature because of the larger grain. For recycled specimens, the ultimate tensile strength and elongation to failure increase with the increasing extrusion temperature in the range of 573~673 K. When the extrusion temperature is higher than 673 K, the ultimate tensile strength and elongation to failure decrease.

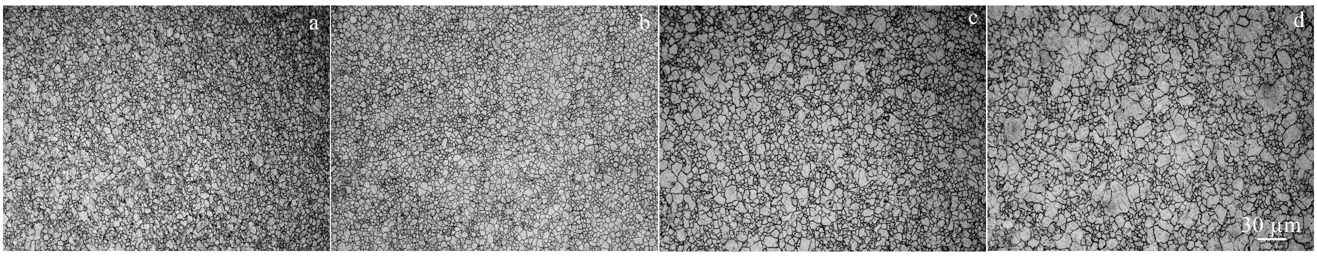

Fig.1 Microstructures of recycled AZ31B magnesium alloy extruded at 573 K (a), 623 K (b), 673 K (c), and 723 K (d)

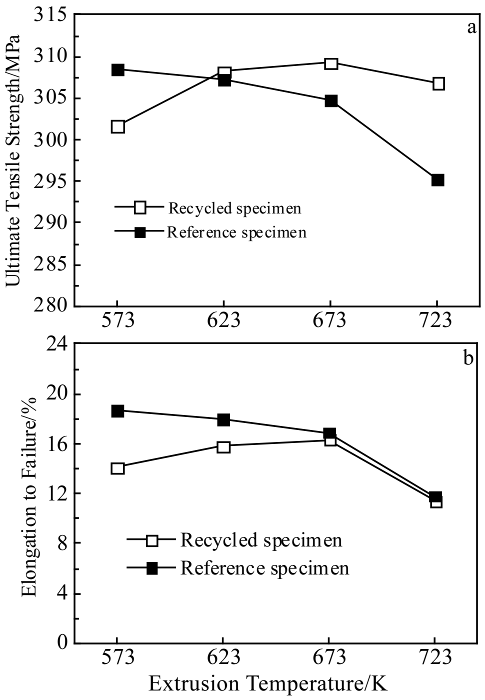

Fig. 2 Mechanical properties of recycled AZ31B magnesium alloy and reference specimen: (a) ultimate tensile strength and (b) elongation to failure

In recycled alloys, chips consolidate each other during hot extrusion, and the shear forces cause chips rearrangement, deformation, destruction of oxide layer and comminuting of individual chips. Enhanced consolidation of chips during solid-state recycling is not only attributed to the physical mechanisms induced by plastic deformation, but also to atom diffusion between chips promoted by severe plastic deformation at higher temperature[14]. Shear deformation at elevated temperatures causes more crystal defects such as dislocation acting as an additive diffusion passage which trigger atom diffusion. High-temperature shear deformation will not only increase the area and proximity of contact but also result in defect structures that promote atom diffusion and enhance bonding of chips. So elevating temperature can enhance the bonding of chips[15], which improves the tensile properties of the recycled materials. When the bonding of chips is strong enough, higher temperature will result in coarse microstructure and deterioration of the mechanical properties. The recycled specimen extruded at 673 K exhibits the highest ultimate tensile strength of 309.2 MPa and the highest elongation to failure of 16.3%.

The recycled specimens show higher ultimate tensile strength than reference specimens when extrusion temperature is above 623 K. Finer grains of recycled specimen can account for its higher strength. Moreover, oxide particle derived from broken oxide film will act as an obstacle of dislocation moving and produce strengthening, but the strengthening effect is expected to be small due to the small amount of oxides in recycled alloy. The recycled alloys show inferior ductility compared with reference alloys.

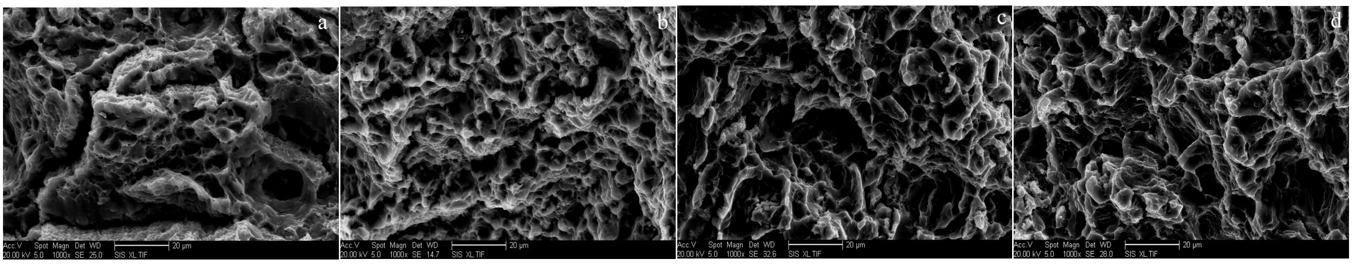

Fig. 3 Fractography of recycled AZ31B magnesium alloy extruded at 573 K (a), 623 K (b), 673 K (c), and (d) 723 K

Fig.3 shows the fracture surfaces of the recycled alloys fabricated at various temperatures. The recycled magnesium alloys exhibit mixed fracture mechanism of quasi-cleavage and microvoid convening fracture. Micro-cracks can be seen on the fracture surfaces of all the recycled alloys. During the tensile test, the uncooperative deformation behaviors of the oxides and matrix cause the stress focus in the matrix around the oxide particles, and the micro-voids or microcracks are prone to forming in this region, which can lead to early fracture through crack growing. The cracks in recycled specimen prepared at 573 K are larger because the cracks form relatively earlier during the tensile test, and the recycled specimen shows lower elongation. Both the number and size of the cleavage planes increase with the increasing extrusion temperature, which is in accordance with the declined elongation for recycled specimen prepared at 723 K.

2.2 Microstructure and mechanical property with different extrusion ratios

Fig.4 shows the microstructures of recycled alloys processed with various extrusion ratios. When the extrusion ratio is 4:1, some large original grains are surrounded by fine recrystallized grains, so the dynamic recrystallization partially occurs. With increasing extrusion

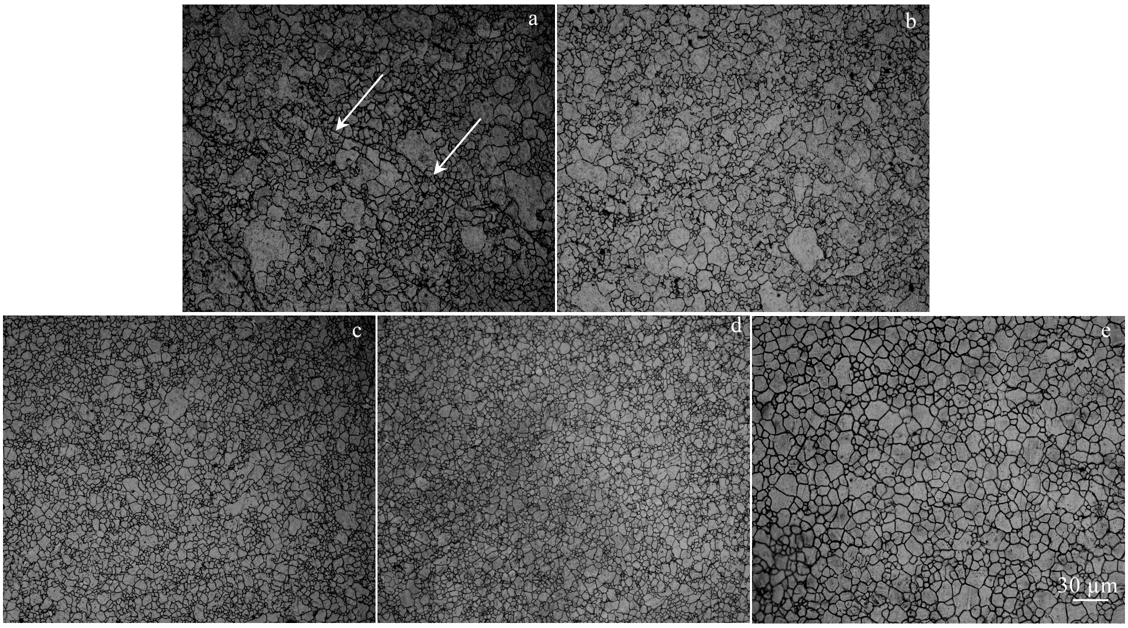

Fig.4 Microstructures of recycled AZ31B magnesium alloy extruded with different extrusion ratios: (a) 4:1, (b) 11:1, (c) 25:1, (d) 44:1, and (e) 100:1

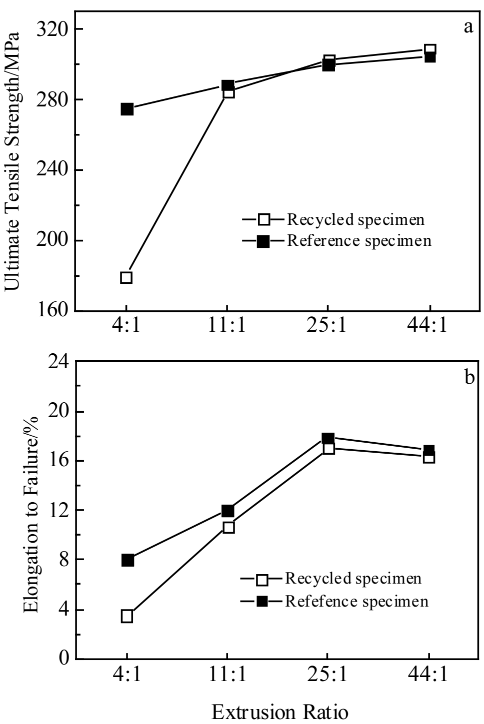

Fig. 5 Mechanical properties of AZ31B magnesium alloy at different extrusion ratios: (a) ultimate tensile strength and (b) elongation to failure

ratio, coarse old grains are taken up by new recrystallized grain gradually. When extrusion ratio increases to 25:1, the alloy almost shows completely recrystallized microstructure with the mean grain size of 9.8 μm. When extrusion ratio reaches 44:1, more uniform, equiaxed-like finer grains are obtained, and the mean grain of size is 7.9 μm.

Large plastic deformation causes severe crystal structure aberrance and an increase of stored energy, and this provides favourable condition for recrystallization. The higher the extrusion ratio is, the greater the potential for the recrystallization nucleating is, and so the smaller the recrystallized grain is[8]. However, when the extrusion ratio increases to 100:1, grain coarsening occurs, and the grain size is 11.4 μm. This may be ascribed to the more heat generated by friction and deformation energy during extrusion. So grain refinement of dynamic recrystallization occurs only in a certain deformation degree.

As shown in Fig.4a, some boundaries between chips can be seen with the low extrusion ratio of 4:1, indicating the poor bonding between chips. When extrusion ratio increases to 11:1, interfaces are dissolved completely, and interface bonding is enhanced with deformation degree increase.

Fig.5 shows the properties of the recycled and reference samples as functions of extrusion ratios. The ultimate tensile strength of the recycled and reference

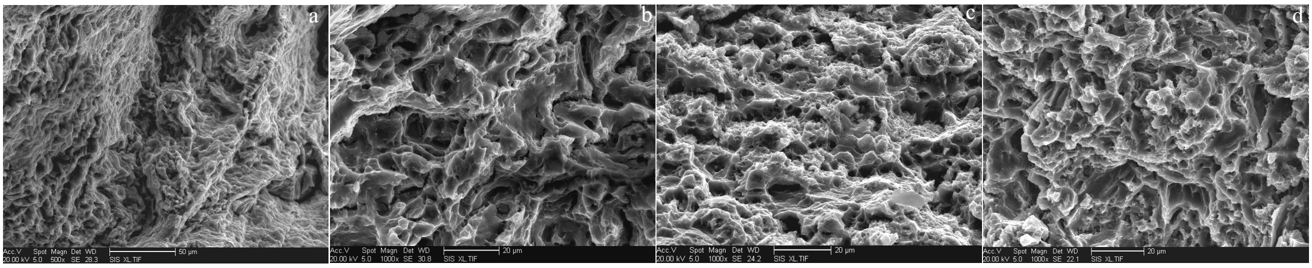

Fig.6 Fractography of recycled AZ31B magnesium alloy extruded with different extrusion ratios: (a) 4:1, (b) 11:1, (c) 25:1, and (d) 44:1

samples increase with increasing extrusion ratio, and it is consistent with the microstructure which becomes finer and more homogeneous. When the extrusion ratio increases to 25:1, the ultimate tensile strength of the recycled specimens is higher than that of the reference specimens.

The interface that acts as the transmitting and sharing of the load will affect mechanical properties of a material. For magnesium alloy recycled by solid state process, interface bonding is another key factor affecting the mechanical properties besides recrystallization microstructure. When the extrusion ratio is high enough, shear stress makes oxide film broken up fully. Shear deformation generates more structure defects and heat energy, and results in the good bonding of chips. Compared with reference specimens, recycled specimens have finer grain, which can account for its higher strength. Whereas the good bonding of chips is the prerequisite for the excellent mechanical properties.

Recycled and reference specimen with the extrusion ratio of 25:1 exhibit the highest elongation to failure of 17.0% and 17.9%, respectively. As described above, grain becomes gradually finer and more uniform when the extrusion ratio increases from 4:1 to 44:1. Grain refinement is the effective means for improving both strength and ductility, and elongation to failure of alloy should increase monotonously with the extrusion ratio increasing. This disagrees with the result in this study. The reason is that deforming still continues when nucleus is growing during the dynamic recrystallization. New dynamic recrystallized grains have a certain degree of strain. As extrusion ratio increases, deformation degree of recrystallized microstructure increases, and the deformation strengthening enhances. When extrusion ratio increases to a certain degree, deformation strengthening results in reduce ductility although grain becomes finer.

Fig.6 exhibits the fractography of the recycled alloys fabricated with different extrusion ratios. From Fig.6a, it can be seen that fracture almost occurs along the bonding surface of chips. Low-deformation degree leads to the weak bonding between chips and the inferior tensile properties consequently. In Fig. 6c, a large number of dimples can be seen, which is in accordance with the high ductility of the alloy with extrusion ratio of 25:1.

3 Conclusions

1) In the investigated extrusion temperature range of 573~723 K, recycled AZ31B magnesium alloys exhibit complete dynamic recrystallization microstructure. Average grain size increases with the increasing extrusion temperature.

2) High-temperature shear deformation can realize sufficient contact of chips and cause crystal defects, which can promote the diffusion bonding between chips. Recycled alloy extruded at a certain temperature shows the excellent tensile property, and this can be attributed to the two contrary effects that include the enhanced bonding condition between chips and increased grain size with the increasing extrusion temperature.

3) Increasing extrusion ratio in a certain range results in the grain refinement. Ultimate tensile strength of recycled alloy increases when extrusion ratio increase from 4:1 to 44:1 because of finer grain and enhanced bonding between chips. Whereas recycled alloy with extrusion ratio of 25:1 have the highest elongation to failure, and further increasing extrusion ratio brings about declined ductility due to deformation strengthening.

References

1 Zhang Q, Li Q A, Chen J *et al*. *Journal of the Chinese Society of Rare Earths*[J], 2015(6): 718 (in Chinese)

2 Xu S W, Oh-Ishi K, Sunohara H *et al*. *Materials Science & Engineering A*[J], 2012, 558(51): 356

3 Lu W U, Pan F S, Yang M B *et al*. *Transactions of Nonferrous Metals Society of China*[J], 2011, 21(4): 784

4 Watanabe H, Moriwaki K, Mukai T *et al*. *Journal of Materials Science* [J], 2001, 36(20): 5007

5 Wang J Y, Lin Y N, Chang T *et al*. *Materials Transactions*[J], 2006, 47(4): 1047

6 Nakanishi M, Mabuchi M, Saito N *et al*. *Journal of Materials Science Letters*[J], 1998, 17(23): 2003

7 Chino Y, Lee J S, Nakaura Y *et al*. *Materials Transactions*[J], 2005, 46(12): 2592
8 Zhao Z D, Chen Q, Yang L *et al*. *Transactions of Nonferrous Metals Society of China*[J], 2011, 21(2): 265
9 Wu S Y, Ji Z S, Zhang T. *Journal of Materials Processing Technology*[J]., 2009, 209(12): 5319
10 Valle J A D, Ruano O A. *Materials Science & Engineering A*[J], 2008, 487(1-2): 473
11 Ion S E, Humphreys F J, White S H. *Acta Metallurgica*[J], 1982, 30(10): 1909
12 Ding H, Liu L, Kamado S *et al*. *Journal of Alloys and Compounds* [J], 2008, 456(1-2): 400
13 Ji Z S, Hong Y, Zhao M. *Transactions of Nonferrous Metals Society of China*[J], 2005(S3): 236
14 Peng T, Wang Q D, Han Y K *et al*. *Journal of Alloys and Compounds*[J], 2010, 503(1): 253
15 Lee J S, Chino Y, Hosokawa H *et al*. *Materials Transactions*[J], 2005, 46(12): 2637

固相再生 AZ31B 镁合金的组织与力学性能

武淑艳[1]，吉泽升[2]，胡 明[1]，黄志求[1]，田春英[1]，吴明忠[1]
(1. 佳木斯大学，黑龙江 佳木斯 154007)
(2. 哈尔滨理工大学，黑龙江 哈尔滨 150040)

摘 要：在不同的挤压温度和挤压比下，将AZ31B镁合金机加屑冷压后热挤压固结而再生镁合金。与铸锭挤压合金对比，从动态再结晶组织与屑间结合情况两个主要方面分析了加工工艺对再生合金力学性能的影响。随着挤压温度升高，再生合金的极限抗拉强度和延伸率先增加而后降低。随挤压温度升高，晶粒长大与屑间结合增强的相反作用共同导致了再生合金力学性能的变化。当挤压比从4:1 增加到44:1，晶粒细化且屑间结合增强，使再生合金的抗拉强度增加。而当挤压比大于25:1时，由于显著的形变强化作用导致延伸率下降。
关键词：固相再生；AZ31B 镁合金；挤压温度；挤压比；力学性能

作者简介：武淑艳，女，1977 年生，博士，佳木斯大学材料科学与工程学院，黑龙江 佳木斯 154007，电话：0451-8618702，E-mail: wsy_hit@sina.com