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ARTICLE

Preparation of High-Performance AZ31 Magnesium Alloy with Bimodal Grain Structure by Single-Pass Hot Rolling

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Abstract: AZ31 magnesium alloy was used as the object of study to fabricate an alloy with the bimodal grain structure using single-pass hot rolling, and to explore how this structure enhances the strength and plasticity of the alloy. The results show that the formation of the bimodal grain structure is more pronounced at rolling temperatures ranging from 350 °C to 450 °C, especially under conditions of large reduction (⩾40%). The optimized proportion and distribution of the bimodal grain structure play a pivotal role in simultaneously enhancing the strength and ductility of the alloy, significantly impacting the mechanical properties. The rolled sheet with the bimodal grain structure achieves an ultimate tensile strength of 258.3 MPa and an elongation of 17.1% under a rolling reduction of 40% with the rolling rate of 75 m/min and rolling temperature of 400 °C. Adjusting rolling parameters, including temperature, reduction ratio and rolling rate, is crucial for optimizing the bimodal grain structure, thereby achieving a balance between plasticity improvement and high strength maintenance.

Key words: AZ31 magnesium alloy; bimodal grain structure; twinning; strength and plasticity enhancement

1 Introduction

Scientists have long sought for metallic structural materials that exhibit both high strength and high plasticity[1–3]. However, alloys typically exhibit a trade-off between these properties: as strength increases, plasticity often decreases[4]. While grain refinement has been recognized as a mean to simultaneously boost strength and plasticity in alloys, and the formation of nanocrystals usually leads to a dramatic decline in plasticity, despite significant gains in strength[5]. Research has identified that fine grains struggle to accumulate dislocations, leading to lower hardening rate and reduced ductility. To address this challenge, a novel bimodal grain distribution has emerged, combining fine and coarse grains in a heterostructure[6]. Such bimodal microstructures have attracted significant attention and yielded impressive outcome with coarse grains facilitating dislocation initiation and enhancing work hardening and plasticity, while fine grains contributing to coordinated plastic deformation and inducing high back-stress hardening effects[7].

Ji et al[8] significantly increased the strength and plasticity of Mg-16Li-2.5Zn-2.5Er alloy by controlling dynamic recrystallization (DRX) and regulating the secondary phase through hot extrusion and cold rolling. Zhu et al[9] enhanced the Mg-5Li-1Al alloy by adding 0.5Nd or 0.5Y and using multi-pass rolling. Zhang et al[10] improved Mg-8Y-1Er-2Zn alloys by controlling DRX and the secondary phase via hot extrusion and aging. Zhang et al[11] modulated the microstructure of Mg-3Al alloys by adding Ca and Gd. During multi-passes hot rolling process, the grains exhibit a bimodal grain structure when the total reduction is 60% with many fine recrystallized grains surrounded by deformed grains that possess twins in the matrix. He et al[12] prepared a bimodal microstructure by a combination of hot forging, extrusion and annealing processes, but the process steps were cumbersome.

Previous studies have shown that most researchers have prepared the bimodal grain structure by extrusion and multiple passes, or by controlling the precipitation of the secondary phase and adding alloying elements. This study focused on the AZ31 magnesium alloy, due to its minimal precipitation phase, aiming to develop the bimodal grain structure through

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a straightforward single-pass hot rolling process, which is simple and easy-to-operate. This approach seeks to enhance both strength and plasticity, exploring the formation, preparation and evolution of bimodal grain structure. The factors controlling the coarse/fine grain ratio and their impact on material properties were investigated, aiming for precise control of the bimodal grain structure.

2 Experiment

The initial material used in this study was a cast AZ31 magnesium alloy sheet with the composition shown in Table 1. The alloy underwent homogenization at 400 °C for 12 h. Subsequently, the alloy was cut into slabs measuring 100 mm (rolling direction) ×60 mm (transverse direction) ×5 mm (normal direction). The rolling was conducted using a double-roll mill with a diameter of 330 mm. These slabs were then rolled with temperatures of 350, 400 and 450 °C. Additionally, the alloy was subjected to varying rolling reductions (20%, 40% and 50%) and rolling rates (20, 40, 55 and 75 m/min) in a single pass. Subsequently, the rolled specimens were air-cooled before conducting optical microscope (OM) observation.

After polishing the specimens using 400#, 800#, 1200# and 2000# sandpapers, a 10vol% nitric acid-alcohol solution was used for chemical polishing for 30 s. The specimens were then etched in a picric acid solution (5 g picric acid+70 mL ethanol+10 mL glacial acetic acid+10 mL water). Then, specimens underwent electrolytic polishing treatment at room temperature through a 37.5vol% phosphoric acid alcohol solution. The voltage for electrolytic polishing was set to 8 V with a duration of 35 s. The room temperature gauge length was conducted on a SANS-CMT 5105 tensile testing machine with the tensile mark of 25 mm and the tensile rate of l mm/min.

3 Results and Discussion

3.1 Initial microstructure

Fig. 1 shows the initial microstructure of AZ31 Mg alloy sheets. The uneroded SEM image in Fig.1a confirms that the secondary phase particles in the microstructure of AZ31 Mg alloy sheet have disappeared after 400 °C/12 h homogenization treatment. Fig.1b shows that the microstructure is mainly composed of coarse and equiaxed grains with an average size of 600 μm, and there are a few twins in the microstructure.

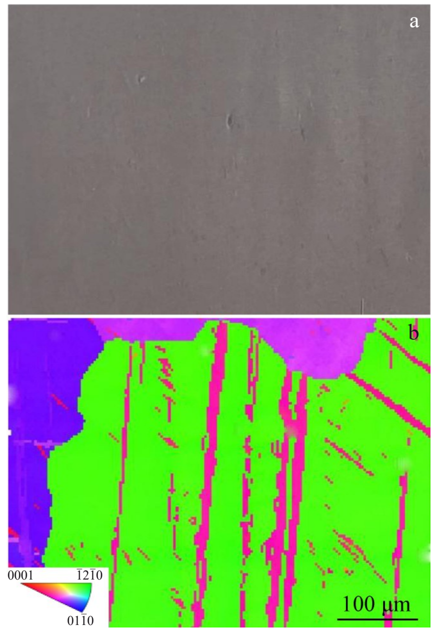

Fig.1 SEM image (a) and IPF map (b) of initial AZ31 Mg alloy sheets

3.2 Effect of process parameters on bimodal grain structure of AZ31 Mg alloy

The bimodal grain structure of the AZ31 Mg alloy is affected by the rolling temperature, rolling rate and rolling reduction. The microstructures resulting from hot rolling between 350 °C to 450 °C with various reduction amounts and rolling rates are displayed in Fig. 2. More bimodal grain structures can be obtained under conditions of large reduction (⩾40%), high rolling rates (⩾55 m/min) and higher temperatures (350–450 °C).

Table 1 Chemical composition of AZ31 Mg alloy sheet (wt%)

Al	Zn	Mn	Fe	Ni	Cu	Mg
2.80	0.88	0.20	0.031	0.001	0.001	Bal.

The findings show that the temperature during the rolling process has a significant effect on both the formation and the proportion of the bimodal grain structure. The rolling temperature is actually lower as the rolls are not heated. When the rolling temperature reaches 350 °C, twinning nucleation becomes the primary mechanism of reduction deformation. The average size of recrystallized grains in the microstructure of the bimodal grain is relatively small. However, as the rolling temperature increases, the original grain size also increases. At the same time, a few recrystallized grains form at the grain boundary, and the sheet rolled at 400 °C exhibits more bimodal grain structures than that rolled at 350 °C. The number of bimodal grain structures decreases after rolling at 450 °C.

Nucleation occurs preferentially within twins and at grain boundaries under conditions of high temperature, large reduction (⩾50%) and high strain rates (⩾55 m/min). Increasing the rolling rate has been found to effectively promote DRX, leading to a higher proportion of fine grains in the bimodal grain structure. The rolling reduction is a crucial factor in creating the bimodal grain structure. A large number of twins arise in the microstructure following rolling when the reduction is small. As the reduction increases, there is a significant increase in the proportion of fine grains in the bimodal grain structure.

3.3 Coordinated reduction behavior of AZ31 Mg alloy with bimodal grain structure

DRX grains with a distinct band-like distribution emerge within the uncrystallized region, resulting from the shear band nucleation and growth in AZ31 Mg alloy, as shown in Fig.2f.

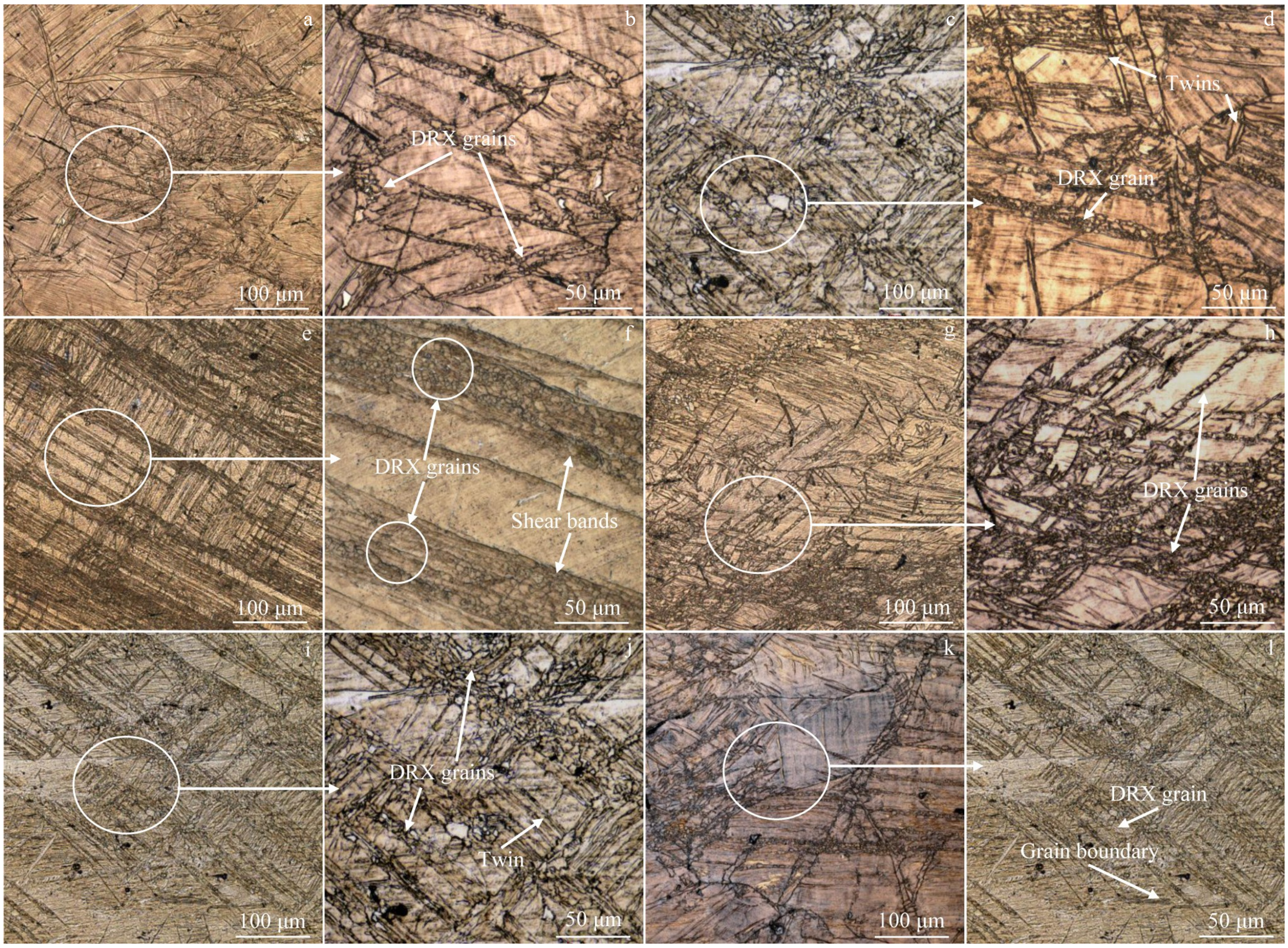

Fig.2 Microstructures resulting from hot rolling between 350 °C to 450 °C with various reduction amounts and rolling rates: (a–b) 350 °C, 50%, 55 m/min; (c–d) 350 °C, 50%, 75 m/min; (e–f) 400 °C, 30%, 40 m/min; (g–h) 400 °C, 50%, 55 m/min; (i–j) 400 °C, 50%, 75 m/min; (k–l) 450 °C, 50%, 75 m/min

During intense deformation processes, like rolling and hot compression, Mg alloys are susceptible to strain localization and shear band formation. The shear bands formed during thermal deformation contain numerous dislocations and stored distortion energy[11]. Under favorable conditions, DRX nucleation and growth occur preferentially within these shear bands, ultimately leading to bimodal grain structures in Mg alloys. The bimodal grain structure of the Mg alloy consists of DRX grains with a band-like distribution and coarse primary grains segmented by the shear bands[13]. Fig. 3 – Fig. 4 are schematic diagrams of nucleation in twins within the bimodal grain structure. The nucleation mechanisms in the bimodal grain structure are primarily twinning and shear band nucleation.

Twins initially form within the original grains during the rolling process, promoting DRX and resulting in the size of recrystallized grain corresponding to the width of the twins[14–16]. The initial stage of rolling induces substantial deformation, resulting in the increase in formation of twin crystals within the material, which is a phenomenon prominently occurring within the original coarse grains[17–19]. An accumulation of numerous dislocations and localized stresses is observed near the twin boundaries, providing

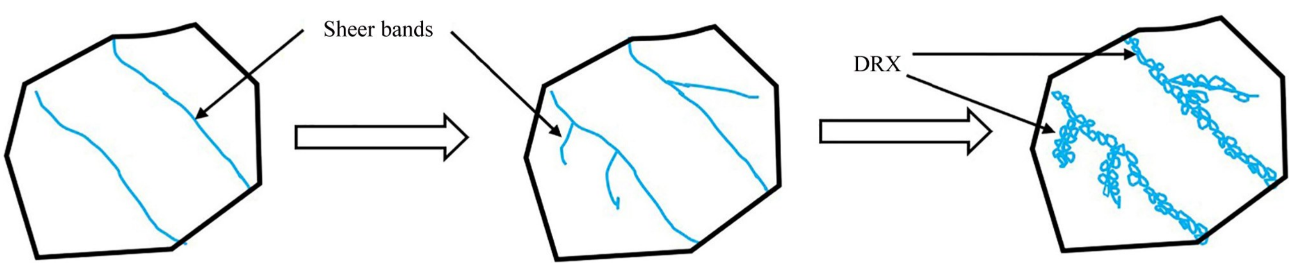

Fig.3 Schematic diagram of shear band nucleation

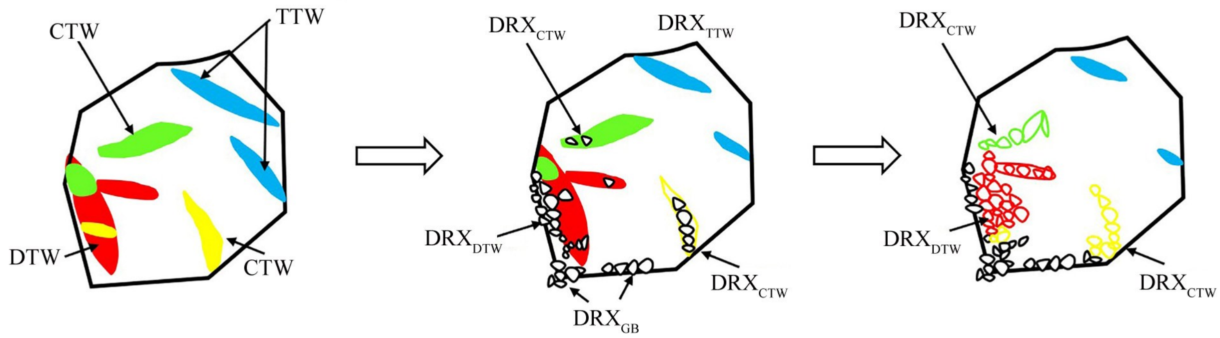

Fig.4 Schematic diagram of nucleation at twins and grain boundaries

favourable conditions for the nucleation of new grains[20].

Concurrently, the low strain rate (≤40 m/min) affords sufficient time for the internal microstructure within the twins to undergo rearrangement. This internal rearrangement facilitates the initiation of DRX nucleation within the twins, as depicted in Fig. 4, in which CTW, TTW and DTW mean compression twin, tension twin and double twin, respectively. Throughout the rolling process, the twins embedded in the original coarse grains progressively undergo DRX, culminating in the formation of nascent DRX particles. These incipient DRX particles incrementally proliferate, eventually saturating the twin structures within the original coarse grains. Within the microstructure of Mg alloy, the finely DRX grains intermingle with the coarse grains and originally deformed grains, separated by the twin boundaries, thereby establishing the bimodal grain structure. The emergence of this bimodal grain structure significantly influences overall performance of the Mg alloy, preserving the strength of the original grains, while endowing it with the toughness characteristics of the DRX grains.

Fig.5 illustrates the impact of various process parameters on the percentage of fine grains in the bimodal grain structure. The combined effects of reduction, rolling rate and temperature influence the proportion of fine grains. Under various rolling conditions, these three factors jointly to influence the formation of the bimodal grain structure. The increase in reduction and temperature is approximately proportional to the increase in percentage of fine grains.

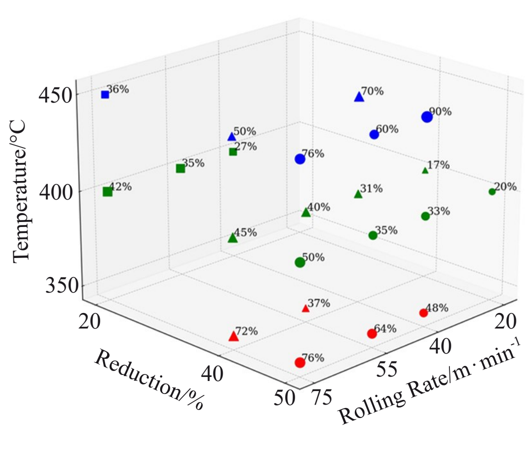

Fig.5 Scatter plot of percentage variation of fine grains in bimodal grain structures with different process parameters

3.4 Mechanical properties of AZ31 Mg alloy with bimodal grain structure

The objective of this study is to elucidate the interrelationship between mechanical properties and microstructural alterations through room temperature tensile experiments on sheets subjected to single-pass rolling. To ensure experimental accuracy, the same sheet underwent tensile testing three times under uniform conditions with the average values of the data used to construct histograms depicting the mechanical properties at varying temperatures (Fig. 6). The labels on the horizontal axes, from a to l, are explained in Table 2. Red squares within the figure highlight the mechanical properties characteristic of sheets with the bimodal grain structure.

In this investigation, the bimodal grain structure obtained at 400 °C, with a 40% reduction in thickness and a rolling rate of 75 m/min is shown in Fig. 7a, exhibiting a tensile strength of 258.3 MPa and an elongation of 17.1%, and demonstrating commendable strength and ductility. When metallic materials with a bimodal grain structure are subjected to plastic deformation, the dislocation density inside fine grains may quickly become saturated. At the same time, coarse grains provide more space for accommodating generated dislocations[16].

Significantly, the grain boundary structure diagram depicted in Fig. 7b elucidates the impact of varying twin types on DRX and bimodal grain structures in magnesium alloys during rolling. Specifically, the green-labeled $\{10\bar{1}1\}$ compression twins and red-labeled $\{10\bar{1}1\}$-$\{10\bar{1}2\}$ secondary double twins exhibit a higher propensity to facilitate DRX compared to the blue-labeled $\{10\bar{1}2\}$ tensile twins. The $\{10\bar{1}1\}$ compression twins and $\{10\bar{1}1\}$ - $\{10\bar{1}2\}$ secondary double twins play a crucial role in the nucleation of new grains, which improves the strength and ductility of Mg alloys[20–21]. This indicates that through the strategic selection of reduction conditions and strain rate, it is possible to selectively induce certain twin types or bimodal grain structures[22–24], thereby optimizing the microstructure and properties of Mg alloys.

At the rolling temperature of 350 °C, the initial stage characterized by a low rolling rate amplifies the reduction

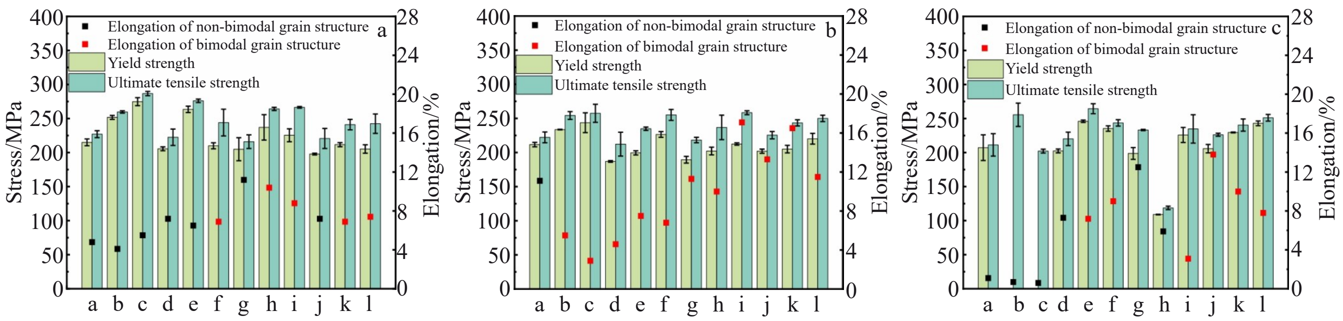

Fig.6 Comparison of mechanical properties of sheets after rolling at different temperatures: (a) 350 °C; (b) 400 °C, and (c) 450 °C

Table 2 Process parameters of different specimens used for tensile test

Specimen	a	b	c	d	e	f	g	h	i	j	k	l
Rolling rate/m·min^{-1}	20	20	20	40	40	40	55	55	55	75	75	75
Reduction/%	20	40	50	20	40	50	20	40	50	20	40	50

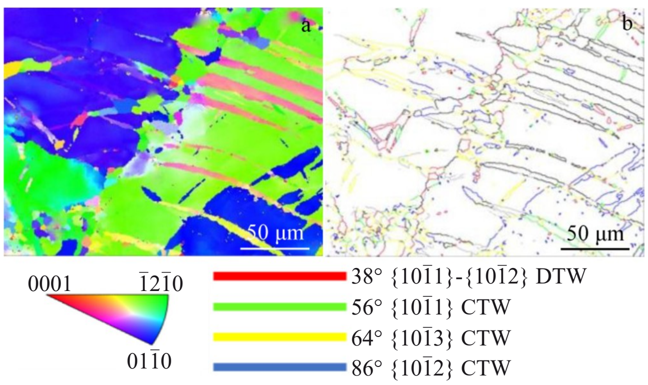

Fig.7 IPF map (a) and grain boundary map (b) of bimodal grain structure with optimal performance

resistance of material during the tensile process, attributable to the extensive presence of twins within the structure, thereby augmenting the strength of the material. However, as the rolling rate increases and the reduction level rises, the grain distribution within the microstructure of rolled sheets becomes more irregular, resulting in a noticeable decline in the strength of sheet, as illustrated in Fig.6a.

At rolling temperature of 400 °C, sheets exhibiting the bimodal grain structure characterized by enhanced strength and plasticity are more commonly produced, particularly under conditions of greater strain and faster rolling rate. In this investigation, the sheets with bimodal grain structure obtained at 400 °C with a 40% reduction in pressure and the rolling rate of 75 m/min exhibit a tensile strength of 258.3 MPa and an elongation of 17.1%, demonstrating commendable strength and ductility. The bimodal grain structure is distinguished by a staggered distribution of fine DRX grains within the twin crystals with the fine grains of approximately 35% to 45% of the structure, as shown in Fig. 7a. The recrystallized grains provide better plasticity, and the twins provide more twin boundaries to impede dislocation motion during reduction, which in turn improves the strength of the material.

At rolling temperature of 450 °C, the prevalence of sheets with the bimodal grain structure decreases compared to that processed at 400 °C, primarily due to the accelerated grain growth associated with the elevated temperature. When rolling reduction is low (⩽40%), the resulting structure predominantly features coarse primary grains and twin crystals with an increased incidence of cracking post-rolling, leading to a marked reduction in material strength and a propensity for brittle fracture.

Future research directions include the understanding of the interplay between reduction conditions and the nucleation/ growth kinetics of the bimodal grain structure, along with exploring the potential of bimodal grain structure to enhance fatigue resistance and other advanced properties.

4 Conclusions

1) There is a direct link between the degree of reduction and the proportion of fine grains in the AZ31 Mg alloy. A higher rolling rate leads to more fine grains. The simplified single-step processing enables the development of the bimodal grain structure, enhancing simplicity and efficiency of process.

2) The optimized bimodal grain structure, achieved at 400 °C with a rolling reduction of 40% and the rate of 75 m/min, yields a tensile strength of 258.3 MPa and elongation of 17.1%, highlighting the effect of the processing strategy in balancing strength and plasticity.

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单道次热轧制备具有双峰晶粒结构的高性能AZ31镁合金

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摘 要：以AZ31镁合金为研究对象，利用单道次热轧制造出具有双峰晶粒结构的镁合金，并探讨该结构如何提高合金的强度和塑性。结果表明，在350~450 ℃的轧制温度下，尤其是在大变形（≥40%）条件下，双峰晶粒结构的形成更为明显。双峰晶粒结构的优化比例和分布在同时提高合金的强度和延展性方面起着关键作用。在轧制变形量为40%、轧制速度75 m/min和轧制温度400 ℃的条件下制备出的轧制板材具有双峰晶粒结构，达到了258.3 MPa的抗拉伸强度和17.1%的延伸率。因此，调整轧制参数，包括温度、变形量和轧制速度，对于优化双峰晶粒结构至关重要，从而实现改善塑性和保持高强度之间的平衡。

关键词：AZ31镁合金；双峰晶粒结构；孪晶；增强增塑

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