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ARTICLE

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

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Abstract: This paper focuses on the AZ31 magnesium alloy, utilizing single-pass hot rolling to fabricate an alloy with the bimodal grain structure, and examines how this structure enhances the alloy's strength and plasticity. The experimental 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 alloy's strength and ductility, significantly impacting the mechanical properties. The rolled sheet with the bimodal grain structure achieved an ultimate tensile strength of 258.3MPa and an elongation of 17.1% under a rolling reduction of 40%, with a rolling rate of 75 m/min and a 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 improved plasticity and maintained high strength.

Key words: AZ31 Mg alloy; Bimodal grain structure; Twinning; Enhanced plasticizing metal

Material scientists have long sought to develop 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 means to simultaneously boost strength and plasticity in alloys, 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 approach has emerged, combining fine and coarse grains in a heterostructured microstructure[6]. Such bimodal microstructures have attracted significant attention and yielded impressive outcomes, with coarse grains facilitating dislocation initiation and enhancing work hardening and plasticity, while fine grains contribute to coordinated plastic deformation and induce 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 and regulating the second 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 dynamic recrystallization and the second 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,

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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 second phase and adding alloying elements. This paper focuses on the AZ31 magnesium alloy, chosen for its minimal precipitation phase,aiming to develop the bimodal-grain structure through a straightforward single-pass hot rolling process, which has the advantage of being simple and easy-to-operate process. This approach seeks to enhance both strength and plasticity simultaneously, exploring the formation, preparation, and evolution of bimodal grain structure. We delve into the factors controlling the coarse/fine grain ratio and its impact on material properties, aiming for precise modulation of the bimodal grain structure.

1 Experiment

The initial material used in this study was a cast AZ31 magnesium alloy sheet with the compositions shown in Table1. The alloy underwent homogenization at 400 °C for 12 hours. Subsequently, the alloy was cut into slabs measuring 100 mm (RD) × 60 mm (TD) × 5 mm (ND). The rolling was carried out using a double-roll mill, which had a diameter of 330mm. These slabs were then rolled with different temperatures:350 °C, 400 °C and 450 °C. Additionally, the alloy was subjected to varying rolling reductions (20%,40% and 50%) and rolling rates (20 m/min,40 m/min,55 m/min and 75m/min) in a single pass. Subsequently, the rolled samples were air-cooled before conducting optical microscopy.

Table 1 Chemical composition of AZ31 magnesium alloy plate (wt%)

Al	Zn	Mn	Fe	Ni	Cu	Mg
2.8	0.88	0.2	0.031	0.001	0.001	Bal.

After polishing the specimens using #400, #800, #1200, and #2000 sandpaper, a 10% nitric acid-alcohol solution was used for chemical polishing for 30 seconds. 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). The specimens underwent grinding by sandpaper and chemical polishing with a 10% nitric acid alcohol solution, followed by electrolytic polishing treatment at room temperature through a 37.5% 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 carried out on a SANS-CMT 5105 tensile testing machine with the tensile mark of 25 mm and the tensile rate of l mm/min.

2 Results and Discussion

2.1 Initial microstructure

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

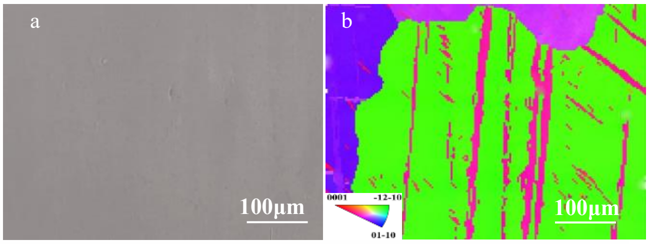


Fig.1 Initial microstructure of AZ31 Mg alloy sheet: (a) the SEM micrograph of uneroded; (b) the IPF maps.

2.2 Effect of process parameters on the bimodal grain AZ31 alloy microstructure

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

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. The mean size of recrystallized grains in the microstructure of the bimodal grain is relatively petite. However, as the rolling temperature increases, the original grain size also increases. At the same time, a few recrystallized grains formed at the grain boundary, and the plate rolled at 400 °C exhibited more bimodal grain structure than the plate rolled at 350 °C. The number of plates with the bimodal grain structure decreased after rolling after rolling at 450 °C.

Nucleation occurs preferentially within twins and at grain boundaries under conditions of high temperatures, large reductions (≥50%), and high strain rates (≥55 m/min). Increasing the rolling rate has been found to effectively promote dynamic recrystallization 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.

2.3 Coordinated reduction behavior of the bimodal grain AZ31 alloy

Dynamically recrystallized grains with a distinct band-like distribution emerged within the uncrystallized region, resulting from the shear band nucleation growth in AZ31 magnesium alloy as shown in Fig.2f. During intense deformation processes like rolling and hot compression, magnesium 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, dynamic recrystallization nucleation and growth occur preferentially within these shear bands, ultimately leading to bimodal structures in magnesium alloys. This structure comprises dynamically recrystallized grains with a band-like distribution and coarse original grains intersected by the shear bands. The bimodal structure of the magnesium alloy consists of dynamically recrystallized grains and coarse primary grains segmented by the shear bands[13]. The Fig.3 and Fig.4 are schematic diagrams of nucleation in twins within the bimodal grain structure. The nucleation mechanism in the bimodal grain structure is primarily mainly twinning and shear band nucleation.

Twins initially form within the original grains during the rolling process, promoting dynamic recrystallization and resulting in a recrystallized grain sizes corresponding to the widths of the twins[14-16].The initial phase of rolling induces substantial deformation, resulting in an increased formation of twin crystals within the material, a structure prominently situated within the original coarse grains[17-19]. An accumulation of numerous dislocations and localized stresses was observed near the twin boundaries, providing favorable conditions for the nucleation of new grains[20].

Concurrently, the low strain rate (≤40 m/min) affords sufficient time for the internal structure within the twin to undergo rearrangement. This internal rearrangement facilitates

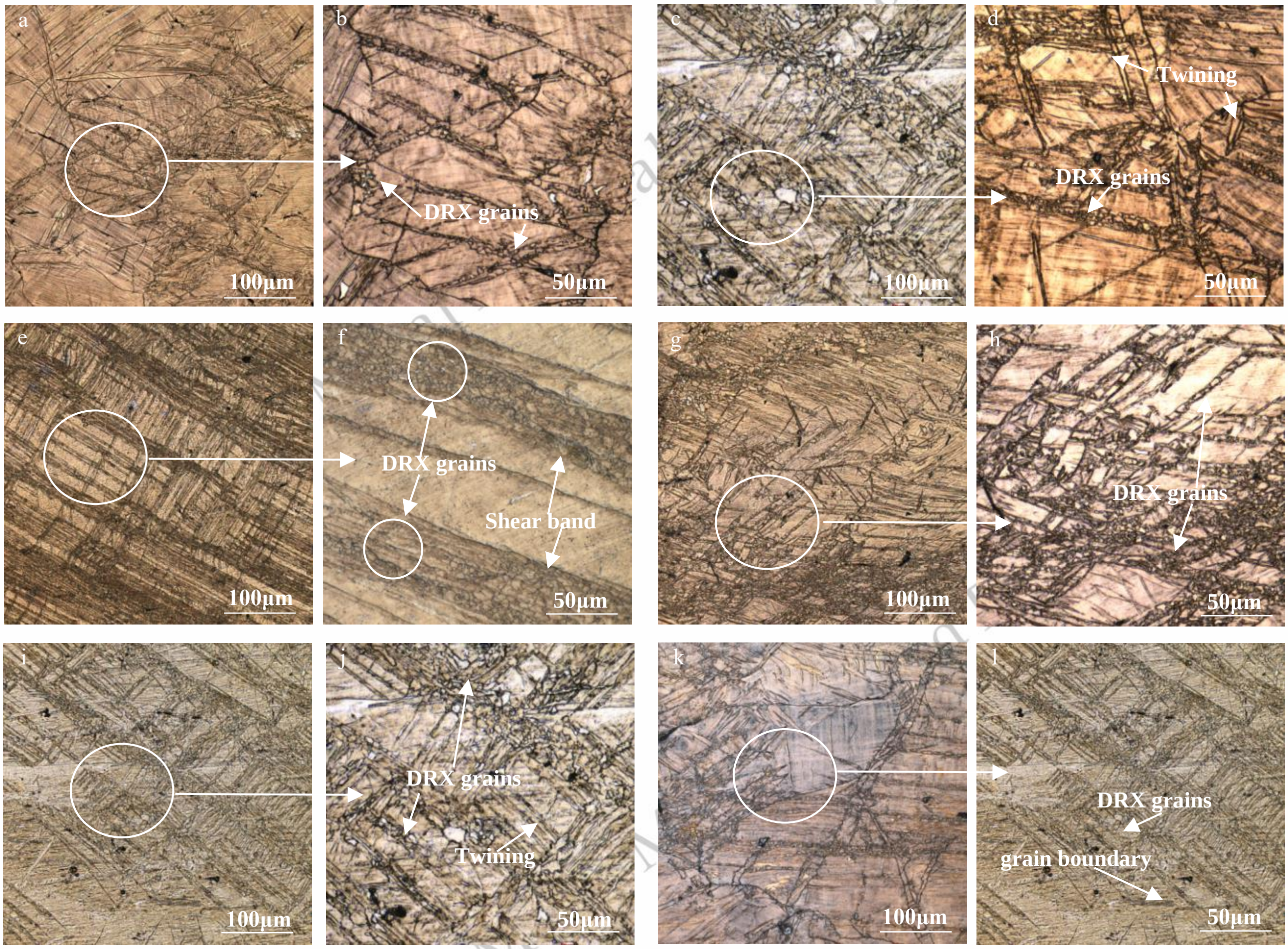


Fig.2 Typical bimodal microstructure: (a,c) 350 °C, rolling reduction 50%,Rolling rate 55 m/min and 75 m/min; (e) 400 °C, rolling reduction 30%,Rolling rate 40m/min; (g,i) 400 °C, rolling reduction 50%,Rolling rate 55 m/min and 75 m/min; (k) 450 °C, rolling reduction 50%,Rolling rate 75 m/min.

the initiation of dynamic recrystallization nucleation within the twins, as depicted in the nucleation schematic presented in Fig.4. Throughout the rolling process, the twins embedded in the original coarse grains progressively undergo dynamic recrystallization, culminating in the formation of nascent dynamically recrystallized particles. These incipient dynamically recrystallized particles incrementally proliferate, eventually saturating the twin structures within the original coarse grains. Within the material's microstructure, the finely dynamically recrystallized grains intermingle with the coarse , originally deformed grains, separated by the twin boundaries, thereby establishing the bimodal structure. The emergence of this bimodal structure significantly influences the material's overall performance, preserving the strength attributes of the original grains while endowing it with the toughness characteristics of the dynamically recrystallized grains.

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

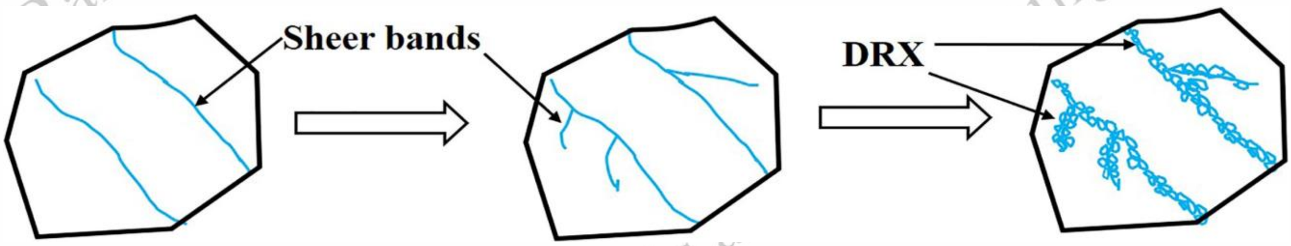


Fig.3 Schematic diagram of shear band nucleation.

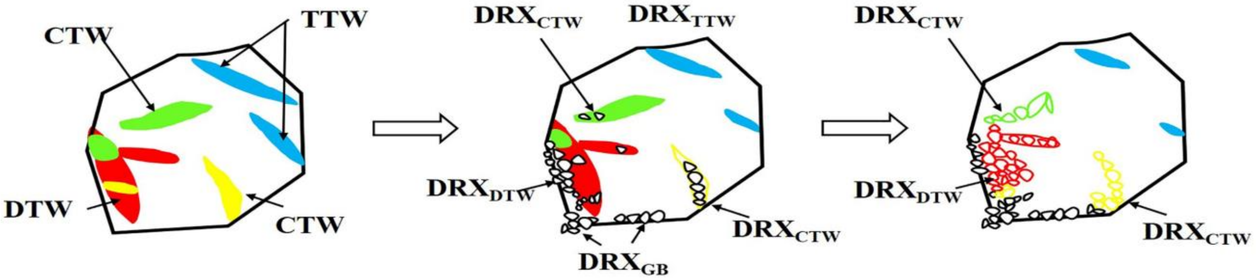


Fig.4 Schematic representation of nucleation at twins and grain boundaries.

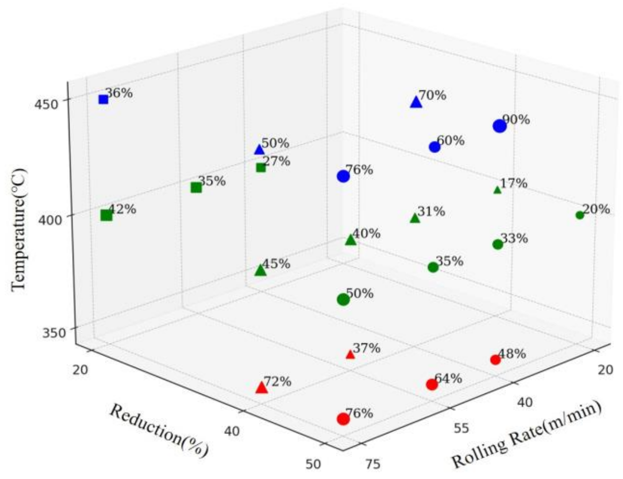
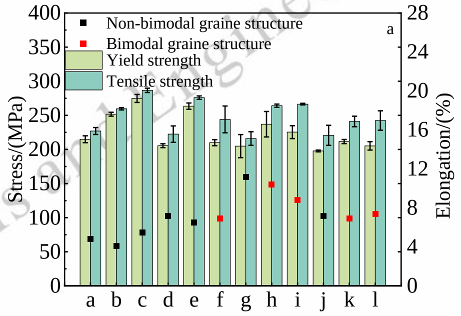


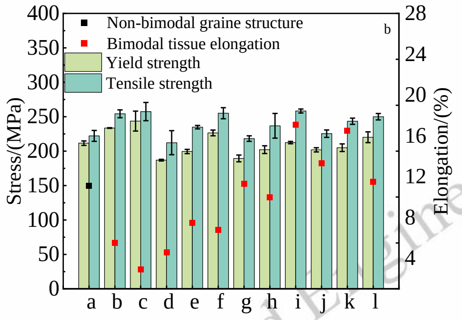
Fig.5 Scatter plot of the variation of the percentage of fine grains in bimodal structures with process parameters.

2.4 Mechanical properties of the bimodal grain AZ31 alloy

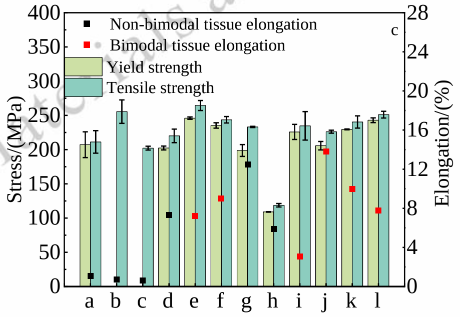
The objective of this study is to elucidate the interrelationship between mechanical properties and microstructural alterations through room temperature tensile experiments on plates subjected to single-pass rolling. To ensure experimental accuracy, the same plate underwent tensile testing three times under uniform conditions, with the mean values of the data used to construct histograms depicting the mechanical properties at varying temperatures (refer to 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 plates with the bimodal grain structure.



Microstructure item after rolling at 350°C



Microstructure item after rolling at 400°C



Microstructure item after rolling at 450°C

Fig.6 Comparison of mechanical properties of plates after rolling with different process parameters: (a) 350 °C; (b) 400 °C and (c) 450 °C.

In this investigation, the bimodal structure processed at 400 °C, with a 40% reduction in thickness, and a rolling rate of 75 m/min, the microstructure is shown in Fig.7a, exhibited a tensile strength of 258.3MPa and an elongation of 17.1%, demonstrating commendable strength and ductility. When metallic materials with a bimodal 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].

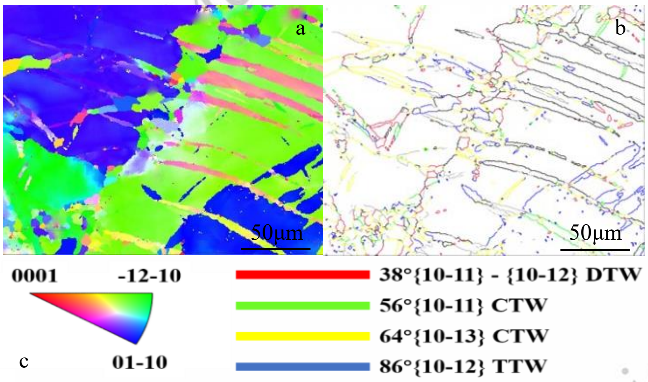


Fig.7 IPF maps of (a) and (b) grain boundary structure maps with optimal performance bimodal grain structure.

Significantly, the grain boundary structure diagram depicted in Fig.7b elucidates the impact of varying twin types on the dynamic recrystallization and bimodal structures in magnesium alloys during rolling. Specifically, the green-labeled {10-11} compression twins and red-labeled {10-11}-{10-12} secondary double twins exhibit a higher propensity to facilitate dynamic recrystallization compared to the blue-labeled {10-12} tensile twins. The{10-11}compression twins and{10-11}-{10-12} secondary double twins play a crucial role in the nucleation of new grains, which improves the strength and ductility of magnesium 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 structures[22-24], thereby optimizing the microstructure and properties of magnesium alloys.

At the rolling temperature of 350 °C, the initial stage characterized by a low rolling rate amplifies the material's reduction resistance during the tensile process, attributable to the extensive presence of twins within the structure, thereby augmenting the material's strength. However, as the rolling rate increases and the reduction level rises, the grain distribution within the rolled plate's structure becomes more irregular, resulting in a noticeable decline in the plate's strength, as illustrated in Fig.6a.

Table 2 Specimen number of tensile test at different temperatures in Fig.6.

Item	a	b	c	d	e	f	g	h	i	j	k	l
Rate (m/min)	20	20	20	40	40	40	55	55	55	75	75	75
Deformation (%)	20	40	50	20	40	50	20	40	50	20	40	50

Under rolling conditions at 400°C, plates exhibiting the bi- -modal grain structure characterized by enhanced strength and plasticity are more commonly produced, particularly under conditions of greater strain and elevated rolling rate. In this investigation, the bimodal structure processed at 400 °C, with a40% reduction in pressure, and the rolling rate of 75 m/min, exhibited a tensile strength of 258.3MPa and an elongation of 17.1%, demonstrating commendable strength and ductility. The bimodal grain structure is distinguished by a staggered distribution of fine dynamically recrystallized grains within the twin crystals, with the fine grains comprising 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 a rolling temperature of 450 °C, the prevalence of plates with the bimodal grain structure decreases relative to those processed at 400 ℃, primarily due to the accelerated grain growth associated with the elevated temperature. When rolling pressure 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.

3 Conclusions

This study successfully demonstrated the fabrication of high-performance AZ31 magnesium alloy with the bimodal grain structure via a streamlined single-pass hot rolling process, offering a novel approach to enhancing the mechanical properties of magnesium alloys. Our findings underscore the critical influence of rolling parameters—specifically, temperature ranging from 350 °C to 450 °C, reduction levels no less than 40%, and moderate to high rolling rates(≥55m/min) —in facilitating the emergence of a bimodal structure that optimizes strength and ductility concurrently.

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

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

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

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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

稀有金属材料与工程杂志社

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摘 要：本文以 AZ31 镁合金为研究对象，利用道次热轧制造出具有双峰晶粒结构的镁合金，并探讨这种结构如何提高合金的强度和塑性。实验结果表明，在 350 ℃至 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 40%、轧制速度 75 m/min 和轧制温度 400 ℃ 的条件下制备出的具有双峰晶粒结构的轧制板材，达到了 258.3MPa 的抗拉强度和 17.1%的延伸率。研究表明：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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