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Effect of twinning and Al–Nd phase on dynamic recrystallization in rolled Mg–Al–Zn–Nd alloy at the moderate strain rate

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Abstract

The dynamic recrystallization of Mg–Al–Zn–Nd alloy during moderate strain rate rolling was studied using electron backscatter diffraction (EBSD) and an energy dispersive spectrometer (EDS). The results showed three kinds of twinnings produced in the alloy in the strain rate range of 4.2 s\(^{-1}\)–7.3 s\(^{-1}\), including \{10\(\overline{1}\)2\}\ extension twinning, \{10\(\overline{1}\)1\}\ contraction twinning, and \{10\(\overline{1}\)1\}\ – \{10\(\overline{1}\)2\}\ double twinning. The extension twinnings decreased gradually with the increase of strain rate. The dynamic recrystallization mechanisms during hot rolling under moderate strain rate conditions mainly include grain boundary nucleation, twinning nucleation, and secondary particle assistant nucleation. The dynamic recrystallization mechanism induced by twinning is mainly \{10\(\overline{1}\)1\}\ – \{10\(\overline{1}\)2\}\ double twinnings. In addition, the strain value near the Al–Nd phase and grain boundary is higher than in grain. The Al–Nd particles in Mg–Al–Zn–Nd alloy play an auxiliary nucleation effect on dynamic recrystallization during hot rolling deformation.

1. Introduction

As one of the lightest engineering structural materials, magnesium alloy has been actively developed and widely used in automobiles, aerospace, electronics, and other military and civil fields [1, 2]. Due to its hexagonal close-packed (hcp) crystal structure and insufficient slip system, its plastic deformation ability is relatively low at room temperature [3–5]. As a complementary deformation mechanism, during the deformation of the magnesium alloy, the twinnings can change the crystal orientation to activate other slip systems, or adjust the c-axis direction strain to compensate the limited-slip system for promoting continuous deformation [6, 7]. In general, the deformation process of magnesium alloy is accessible under the condition of low strain rate and high deformation temperature, which can avoid the formation of cracks in the deformation process. At present, people use compression, forging, and other forming methods to realize the deformation of magnesium alloy under the condition of a relatively high strain rate (10 ~ 100 s\(^{-1}\)). Extensive twinnings and subsequent dynamic recrystallization structures are produced in the deformed magnesium alloy. The formation of twinnings and dynamic recrystallization plays a positive role in refining the microstructure, reducing the local dislocation density, releasing the accumulated strain energy, and improving the plasticity of the alloy [8–14]. A lot of meaningful work has been carried out on the effect of twinnings on recrystallization behavior in wrought magnesium alloys. It is considered that twinnings can induce dynamic recrystallization. Liu [15] et al studied twinnings and dynamic recrystallization in the compression process of AZ31 magnesium alloy during a high strain rate (>10 s\(^{-1}\)). The results show that twinnings in the alloy include \{10\(\overline{1}\)2\}\ extension twinning, \{10\(\overline{1}\)1\}\ compression twinning, and \{10\(\overline{1}\)1\}\ – \{10\(\overline{1}\)2\}\ double twinning, and the twin boundary with additional strain energy can induce dynamic recrystallization. Wu [16] et al studied the microstructure of AZ31 magnesium alloy forged repeatedly at a high strain rate (~100 s\(^{-1}\)), and the results showed that high-density \{10\(\overline{1}\)2\}\ and \{10\(\overline{1}\)1\}\ – \{10\(\overline{1}\)2\}\ twinnings and subsequent twinnings could induce dynamic recrystallization (DRX). However, at high strain rates, due to magnesium alloy’s high degree of structural strain, it isn’t easy to obtain direct evidence of the relationship between twinning types and dynamic recrystallization in the literature.

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mentioned above. Therefore, this work performed single-pass rolling deformation of magnesium alloy at a moderate strain rate, hoping to obtain the microstructure characteristics of different twinning types and recrystallization coexisting, so as to help clarify the internal relationship between twinnings and dynamic recrystallization behavior, this is of great significance for deep understanding and enriching the dynamic recrystallization mechanism of wrought magnesium alloys.

As we all know, the rare earth compounds in magnesium alloys have the characteristics of high melting point, hard brittleness, and so on. Therefore, these rare earth compounds can hinder the dislocation movement during the deformation of the magnesium alloy and form stress concentrations around them. The large rare earth compounds are easy to initiate microcracks as crack sources, thus consuming part of strain energy storage. In addition, the study shows that rare earth compounds also have the potential advantage of promoting recrystallization nucleation. Therefore, rare earth compounds also play a positive role in improving the plasticity of the alloy. The authors [21, 22] studied the microstructure evolution and mechanical properties of Mg–Al–Zn–Nd casting alloy in the early stage. They found small-sized high-melting Al–Nd compounds in the alloy, which can hinder the dislocation movement and play the role of secondary phase strengthening. These high melting points Al–Nd compounds are bound to have a specific effect on the microstructure of magnesium alloy during deformation. At present, there are few reports on the impact of Al–Nd compounds on recrystallization in the deformation process of AZ magnesium alloy. Therefore, this article takes the moderate strain rate single-pass rolling deformation Mg–Al–Zn–Nd alloy as the object to study the effects of twinnings and Al–Nd compounds on the dynamic recrystallization of magnesium alloy at different strain rates, which provides the experimental basis for enriching dynamic recrystallization mechanism of wrought magnesium alloy.

2. Experimental procedure

The composition of the experimental as-cast Mg–Al–Zn–Nd alloy studied in this work is shown in Table 1. A sheet with 50 mm × 20 mm × 5 mm was cut from the ingot by wire cutting for a subsequent rolling test. To eliminate the nonequilibrium microstructure, and dissolve the Mg17Al12 phase into the matrix completely of the as-cast alloy, a two-step homogenization (260 °C for 1 h, and 430 °C for 12 h) was conducted, and then water quenching was carried out. After being heated at 400 °C for 15 min, the homogeneous sheets were rolled to different thicknesses by a single pass. The corresponding strain rates are 4.2 s⁻¹, 5.9 s⁻¹, and 7.3 s⁻¹, respectively. The strain rate ε is calculated by the equation of \( \frac{H - h}{H} = \frac{v}{\sqrt{R_1 R_2}} \). Where \( H \) is the initial thickness of the sheet (\( H = 4.4 \text{ mm} \)), \( h \) is the thickness of the sheet after rolling (\( h = 3.96 \text{ mm}, 3.52 \text{ mm} \) and \( 3.08 \text{ mm} \)), respectively, \( v \) is the circumferential rolling speed (\( v = 224.62 \text{ mm s}^{-1} \)), \( R_1 \) is the rolling radius (\( R = 65 \text{ mm} \)).

The samples for EBSD analysis were taken from the RD × TD surface of rolled sheets. The microstructure of the rolled alloy was characterized by QUANTA FEG650 scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). The electron backscatter diffraction (EBSD) data of the alloy were analyzed by Channel 5 software. The acceleration voltage of EBSD data acquisition is 20 kV, and the scanning step is 0.2 ~ 1 μm. The EBSD samples were prepared using AC2 electrolytic polishing solution at −30 °C and 20 V voltage for 60–120 s.

3. Results and discussions

3.1. Initial microstructure

Figure 1 shows the microstructures of as-cast alloy before and after homogenization. As shown in figure 1(a), many coarse Mg17Al12 phases are distributed at the grain boundaries and then dissolved in the matrix after homogenization (figure 1(b)). In our previous study, the residual precipitate phase particles should be rare-earth-containing phases [22, 24].

Figure 2 shows the microstructures of the homogenized Mg–Al–Zn–Nd alloy. As shown in figure 2(a), the alloy’s microstructure shows equiaxed grains, and the inverse pole figure (IPF) map shows that the alloy presents a relatively random orientation distribution. Figure 2(b) shows that the average grain size is about 65 μm. In addition, SEM micrograph and EDS of the alloy after homogenization (figures 2(c), (d)) show that many granular and rod-like Al–Nd phases are distributed in the alloy, and the Mg17Al12 phases have been dissolved completely.

**Table 1. The composition in wt% of alloy.**

| Element | Al | Zn | Nd | Mg |
|---------|----|----|----|----|
| Content | 8.07% | 0.53% | 1.36% | Bal. |

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3.2. Microstructure analysis of rolled alloy

Figure 3 provides the EBSD data of as-rolled Mg–Al–Zn–Nd alloy at the strain rates of 4.2 s\(^{-1}\), 5.9 s\(^{-1}\), and 7.3 s\(^{-1}\). As shown in figures 3(a)–(d), and (g), it can be seen from the IPF map that the \{0001\} basal plane of the most grains have a vertical relationship with the ND direction, so there is an apparent basal texture in the rolled sheet. In addition, it is particularly pointed out that the black particles in figures 3(a)–(d), and (g) are Al–Nd rare earth phases, which distribute at the grain boundaries and inside the grains. Figures 3(b)–(h) show the EBSD maps of the alloy inserting twinnings and DRX. The angular deviation of identifying twinning boundaries is limited to 5\(^\circ\). Then three common twinning boundaries, \{10\(\overline{1}\)2\} 86\(^\circ\), \{10\(\overline{1}\)1\} 56\(^\circ\), and \{10\(\overline{1}\)1\} – \{10\(\overline{1}\)2\} 38\(^\circ\), can be observed, highlighted by red, blue and yellow lines on the figure, respectively. From figures 3(b)–(h), it can be found that there are significant differences in the morphology of twinnings at each strain rate, such as that the shape of \{10\(\overline{1}\)2\} extension twinning is approximately lenticular, while the shape of \{10\(\overline{1}\)1\} – \{10\(\overline{1}\)2\} double twinning is fine to strip. Simultaneously, according to figures 3(b)–(h), it can also be found that the dynamic recrystallization grains are produced within the twinnings and nearby the Al–Nd rare earth phases. The randomly distributed dynamic recrystallized grains are highlighted in teal color on the maps. As shown in figures 3(a), (b), when the strain rate is 4.2 s\(^{-1}\), the dynamic recrystallization grains are mainly distributed near the grain boundary and the rare earth phase, and few twin-induced
recrystallized grains have been found. When the rolling strain rate increases to 5.9 s$^{-1}$ and 7.3 s$^{-1}$, the dynamic recrystallization coincides in grain boundaries and twinnings. Figures 3(d)–(h) show the average kernel misorientation (KAM) maps of the rolled alloy. Low-angle grain boundaries (LAGBs, $2^\circ$–$15^\circ$) and high-angle grain boundaries (HAGBs, $>15^\circ$) are marked by gray lines and black lines on the figures, respectively. It can be seen from KAM maps that the distribution of LAGBs is mainly concentrated at the grain boundaries, and the density of LAGBs increases with the increase of the rolling strain rate. Generally, the presence of LAGBs is beneficial to promote dynamic recrystallization and ultimately convert to HAGs [25, 26]. In addition, it can be further found that the KAM value is higher at the grain boundaries than that in grains. However, the KAM values are lower in most dynamic recrystallized grains, which confirms that the strain value is higher near the grain boundaries, conducive to promoting dynamic recrystallization nucleation.

3.3. Effects of twinning on dynamic recrystallization

Figure 3 shows three kinds of twinnings in the rolled alloy, including \{10\,\overline{1}2\} extension twinnings, \{10\,\overline{1}1\} contraction twinnings, and \{10\,\overline{1}1\} $\rightarrow$ \{10\,\overline{1}2\} double twinnings. In order to understand the relationship between twinning and dynamic recrystallization, the twinning fraction obtained from figure 3 is analyzed, as shown in figure 4. The results show that with the increase of strain rate, the fraction of extension twinnings decreased gradually. When the strain rate is 5.9 s$^{-1}$, the contraction twinning and double twinning fractions have apparent superiority. According to the research, \{10\,\overline{1}2\} twinnings mainly occur in the early deformation stage and act as a slip system, which can coordinate the deformation [27, 28]. However, \{10\,\overline{1}1\} contraction twinnings are mainly excited in the later deformation stage and relieve the stress concentration, which \{10\,\overline{1}1\} usually causes the basal to rotate to the favorable sliding direction [29, 30]. As shown in figure 4, the contraction twinning fraction is relatively tiny at each strain rate. This is because more \{10\,\overline{1}1\} $\rightarrow$ \{10\,\overline{1}2\} double twinnings transformed from the initial \{10\,\overline{1}1\} contraction twinnings during the rolling process [29]. Therefore, \{10\,\overline{1}2\} extension twinning or \{10\,\overline{1}1\} $\rightarrow$ \{10\,\overline{1}2\} double twinning may be the primary twinning type that is dynamic recrystallization.

In addition, the regions of the yellow box in figures 3(b)–(h) have been further analyzed and characterized to confirm further the types of twinning which affect dynamic recrystallization, as shown in figure 5. Figures 5(a)–(c) show that the deformed twinnings recrystallization at all the strain rates. Figures 5(g)–(i) show \{0001\} and
The results show that the misorientation angle between the twinnings and matrix is $38^\circ$, and the common rotation axis is $\{11\bar{2}0\}$. Based on the orientation characteristics, the twinnings shown in figures 5(a)–(c) are double twinnings with an axis angle pair of $38^\circ$ ($\{11\bar{2}0\}$). In particular, the orientations of non-recrystallized twinnings and twin-induced recrystallized grain bands in figures 5(a)–(c) are very close, indicating that they belong to the same twinning before recrystallization. In addition, from figures 5(a)–(c), it can be seen that the orientation of the recrystallized grains in the DRX band is relatively random. Al-Samman [8] and Basu [24] et al. believed that the orientation...
characteristics of twin-induced recrystallization grains seem to be related to the orientation characteristics of initial twinnings. Figures 5(d)–(f) show the uncorrelated (orientation difference between any two points in the test area) angle analysis of the recrystallized grains and matrix in the DRX bands. It is found that the recrystallized grain misorientation angle is concentratedly distributed in the range of 38° ± 5°. Therefore, it is determined that {1011} {1012}-double twinning is the main twining type that affects dynamic recrystallization. The formation of dynamic recrystallization introduced by {1011} {1012}-double twinnings can be attributed to the fact that the stacking faults are facilitated by double twinning, which can promote the formation of LGBs, transform into the HGBs, and then lead to dynamic recrystallization [31].

3.4. Effect of Al–Nd rare earth phase on dynamic recrystallization

Besides, the results in figure 3 show that the dynamically recrystallized grains should have some relationship with Al–Nd rare earth phases more or less. Therefore, to explore the effect of the Al–Nd rare earth phase on dynamic recrystallization, the micro-region EBSD and EDS characterization of the black box regions in figures 3(a)–(g), which contain the Al–Nd rare earth phases have been conducted as shown in figure 6. EDS maps of Al and Nd elements confirm that the bright points are Al–Nd phases. It can be seen from figure 6 that there are some recrystallized grains distributed near or surrounded the Al–Nd rare earth phases. It is also found that there is a considerable KAM value near the Al–Nd rare earth phases, as shown in figures 6(d)–(l). Since the Al–Nd rare earth phases may become an obstacle to the movement of dislocations, resulting in more dislocations accumulating here to generate the strain concentration as the deformation continues. Hence, Al–Nd rare earth phases play an assistant nucleation effect of dynamic recrystallization.

4. Conclusions

In this paper, the effects of twinning and Al–Nd phase on dynamic recrystallization of rolled Mg–Al–Zn–Nd alloy at moderate strain rates were studied. The conclusions are summarized as follows:
(1) At the three strain rates of 4.2 s\(^{-1}\), 5.9 s\(^{-1}\), and 7.3 s\(^{-1}\), there are three twinning types of \{10\overline{1}2\} extension twinning, \{10\overline{1}1\} contraction twinning, and \{10\overline{1}1\} \rightarrow \{10\overline{1}2\} double twinning in the rolled alloy, and the proportion of extension twinning decreases gradually with the increase of strain rate.

(2) The dynamic recrystallization mechanism of Mg–Al–Zn–Nd alloy during hot rolling mainly includes grain boundary nucleation, twinning nucleation and secondary particle assisted nucleation. Among them, the dynamic recrystallization mechanism induced by twinning is mainly \{10\overline{1}1\} \rightarrow \{10\overline{1}2\} double twinning, and the Al–Nd phase plays an auxiliary nucleation role in dynamic recrystallization during hot rolling deformation.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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