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Trans. Nonferrous Met. Soc. China 22(2012) 2066-2071

Transactions of Nonferrous Metals Society of China

www.tnmsc.cn

Hot deformation behavior of AZ91 magnesium alloy in temperature ranging from 350 °C to 425 °C

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Received 17 November 2011; accepted 1 February 2012

Abstract: The flow behavior and microstructure evolution of AZ91 magnesium alloy during a thermomechanical process, hot compression test, was investigated. The specimens were hot compressed at a temperature ranging from 350 °C to 425 °C and at strain rate of 0.1 s^{-1} to the strains of 0.3, 0.5 and peak. Microstructural evolutions were studied using optical and scanning electron microscopes. The results show that during the compression process, the recrystallized grains nucleate along the pre-existing grain boundaries. The amount of dynamically recrystallized grains is increased with strain in a sigmoid scheme followed by Avrami equation. The size of dynamically recrystallized grains also increases at the beginning and decreases after reaching the maximum value.

Key words: AZ91 alloy; hot compression; microstructure evolution; recrystallization; peak strain

1 Introduction

Owing to their low density, high specific strength and good thermal and electrical conductivities, magnesium alloys have been used for a wide variety of applications [1–3]. Improving the fuel efficiency of vehicles and reducing CO_2 emissions due to their high specific strength and stiffness have nominated them as a great alternative for steel and aluminum alloys in the transportation industries [2,4–6]. Also, magnesium alloys can potentially be used instead of plastics in the electronic and computer industries [7].

However, these alloys have not been used for high performance applications due to their low mechanical properties at room and elevated temperatures [8]. The poor formability of magnesium and its alloys at room temperature has limited their applications [8–12]. The restricted formability is due to the lack of independent slip systems at room and ambient temperatures (200–450 °C) [4,12].

Among cast Mg alloys, AZ91 alloy is the most widely used alloy due to its good combination of high strength at room temperature, good cast ability and excellent corrosion resistance [13,14]. However, as it is a

cast alloy, there are limited studies on the microstructure evolution and hot deformation behaviour of AZ91 alloys [13,15]. During the hot deformation of Mg alloys, according to the stacking fault energy (SFE) in Mg $(\gamma_{SF}=125 \text{ mJ/m}^2)$ [11], dynamic recovery (DRV) is expected to be the dominant softening mechanism [12]. However, it has been shown that the main restoration phenomenon is dynamic recrystallization (DRX) due to the lack of easy and active slip system [16,17]. The operation of DRX is of particular importance as it reduces the flow stress and grain size. As there is little data considering the relation between DRX grain size and thermomechanical behaviour of cast Mg alloys [4,12], the present study was designed to investigate the evolution of microstructure during the hot deformation process of AZ91 alloy. To this aim, hot compression tests were carried out over the temperature range of 350-425 °C and DRX features in the resulting microstructures were studied using optical and scanning electron microscopes.

2 Experimental

The experimental material used in the present study was as-cast AZ91 magnesium alloy (Mg-9.2%Al-

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0.8%Zn-0.22%Mn). Homogenization heat treatment was conducted at 420 °C for 24 h followed by water quenching. The hot compression specimens were prepared in the form of cylinders with 15 mm in height and a height to diameter ratio of 1.5. The hot compression tests were done as follows.

1) The specimens were heated up to test temperatures in the range of 350-425 °C.

2) The specimens were soaked for 3 min to be homogenized.

3) Hot compression tests were performed under strain rate of 0.1 s⁻¹ and two separate true strains: i) ε =0.3, ii) ε =0.5.

4) The specimens were water quenched for less than 5 s to preserve as-deformed microstructure.

The tests were done using a Zwick/Roll 25-Ton machine equipped with an electrical resistance furnace, which can maintain temperature variation of ± 5 K. Teflon tape was also used to protect samples from oxidation. Deformed specimens were conducted by standard sample preparation techniques followed by immersion etching in an acetic-picric solution. Scanning electron microscope (SEM) and optical microscope (OM) equipped with an image analyzer were employed for microstructure examination.

3 Results and discussion

3.1 Initial microstructure

Figure 1(a) shows that the initial microstructure of



Fig. 1 SEM images showing microstructures of as-cast ingot (a) and homogenized specimen (b)

the alloy consisted of supersaturated α -Mg as main phase and eutectic $\alpha+\beta$ as minor constitution. The latter was observed at inter-dendritic arms. In order to dissolve β (Mg₁₇Al₁₂) precipitates, the samples were homogenized at 420 °C for 24 h and then water quenched. Figure 1(b) shows the microstructure after homogenization process, containing coarse equiaxed grains with mean diameter of ~200 µm with fine β precipitates at distributed boundaries. The dendritic structure has also disappeared.

3.2 Stress—strain curves

True stress — strain curves obtained during hot compression tests at 350, 400 and 425 °C and strain rate of 0.1 s⁻¹ are presented in Fig. 2. At the initial stage, the stress increases rapidly due to work hardening resulting from the continuous accumulation of dislocation. By increasing the strain, flow stress increases up to a maximum value and thereafter decreases to a steady state. Such flow softening behavior is observed after a critical strain and is generally attributed to the dynamic recrystallization (DRX) phenomenon as well as the nucleation and coarsening of β precipitates [16,18,19]. This behavior is dependent on the deformation temperature.



Fig. 2 True stress—strain curves of deformed samples at strain rate of 0.1 s^{-1} and different temperatures

The peak points of the curves shift to the lower stresses and strains by increasing the deformation temperature, as shown in Figs. 3(a) and (b), respectively. It is well documented that increasing the deformation temperature of Mg alloys leads to decreasing critical resolved shear stresses (CRSS) for slip systems, pyramid and prismatic planes [18]. Consequently, these result in more mobility of dislocations on the slip planes, which are manifested by decreased peak stress. Nucleation of newly recrystallized grains, on the other hand, is effective in decreasing the peak stress by decreasing the dislocations density [19].

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3.3 Evolution of microstructures

The evolution of microstructures at three points of the flow curve, i.e., peak strain, strains of 0.3 and 0.5, is depicted in Fig. 4. At peak strain, dynamically recrystallized grains nucleate at original grain boundaries, forming a necklace grain structure at all tested temperatures. However, the size of recrystallized grains increases and their volume fraction decreases by increasing testing temperature from 350 to 425 °C. Increasing the strain to 0.3 is accompanied by increasing the volume fraction of DRX and finer grains are developed at lower temperatures. This can be observed more clearly in Fig. 5, where the dependence of average grain size is plotted vs strain. It may be seen that at a constant strain, the average grain size of DRX increases with increasing the deformation temperature due to the increase in the restoration rate [16]. Recrystallized grains at the peak point start to grow by increasing strain and the rate of increase of the grain size from peak strain up to 0.3 is relatively equal in all testing temperatures. Increasing strain beyond 0.3, up to 0.5, develops the volume fraction of DRX toward full recrystallization. However, even at the strain of 0.5, there are still some unrecrystallized regions in the microstructure.



Fig. 3 Dependence of peak stress (a) and peak strain (b) to deformation temperature in hot compression experiments



Fig. 4 Microstructure evolution of AZ91 during hot compression tests at temperatures of 350 °C, 400 °C and, 425 °C and strains: (a1), (a2), (a3) Peak strain; (b1), (b2), (b3) ε =0.3; (c1), (c2), (c3) ε =0.5



Fig. 5 Evolution of dynamic alloy recrystallized grain size with true strain at a strain rate of 0.1 s^{-1}

Figure 5 shows the changes in the recrystallized grain size against strain in tested temperatures. The average recrystallized grain size does not follow the same rate of increase as before, and it shows an even decreasing trend at 350 °C. This is a very interesting finding because straining beyond the peak point has been able to continuously decrease DRX grain size. Hot compression flow curves at 400 and 425 °C (Fig. 2) show that the strain of 0.3 is approximately the beginning point of steady state region. Therefore, it is expected to see no noticeable change in DRX grain size by more straining, which is in agreement with the measurements of average grain diameters shown in Fig. 5. However, at 350 °C the steady state is not attained even at the strain of 0.5 and the average size of dynamically recrystallized grains still continues to decrease.

3.4 Volume fraction of recrystallization

Figure 6 shows the volume fraction of dynamically recrystallized grains vs strain at three deformation temperatures which was measured using image analysis technique. It is seen that the general trend of increasing volume fraction follows a well-known S-type growth rate. Moreover, the recrystallization volume fraction is increased by raising the deformation temperature. In general, dynamically recrystallized fraction can be well described by the Avrami equation [16]:

$$X_{\rm DRX} = 1 - \exp\left[-0.3 \left(\frac{\varepsilon - \varepsilon_{\rm c}}{\varepsilon_{\rm p}}\right)^{m'}\right]$$
(1)

where ε_c is the critical strain; ε_p is the peak strain; m' is a material constant [20] which indicates the recrystallization kinetics and depends on the deformation temperature. It is well known that the driving force of DRX decreases as the deformation temperature increases, leading to a hindrance effect on DRX. This leads to a

decrease in the value of m'. Raising the deformation temperature increases the rate of restoration phenomena. In the present study, the amount of m' was found to be 1.4 using linear regression method. The amount of m' for AZ31 alloy was previously reported to be 1.2 [21–24] which is lower than the current value for AZ91. In AZ91 alloy, Al atoms play a pining role on dislocation motion while fine precipitates act as nucleation sites for newly recrystallized nuclei [25,26]. These cause an increasing rate of DRX. Moreover, it has been shown that differences in the initial texture and orientation of basal and prismatic planes during deformation, lead to a change in DRX kinetics [26,27].



Fig. 6 Evolution of dynamically recrystallized fraction with true strain at strain rate of 0.1 s^{-1}

3.5 Recrystallization mechanism and comparison between the present work and other results

As AZ91 is a cast alloy, little study has been performed on its hot deformation behavior. In the present work, our findings on AZ91 were compared with previously published data on AZ31.

Microstructures in the peak strain at the temperatures of 350, 400 and 425 °C (Fig. 4) indicate the formation of newly recrystallized grain at the primary boundaries which clearly shows the occurrence of dynamic recrystallization (DRX). The formation of recrystallization grains can be done with different mechanisms [28]. In magnesium alloys, different mechanisms have been proposed, among which the most common mechanism is discontinuous recrystallization (DDRX). Other mechanisms, including continuous dynamic recrystallization (CDRX), twin induced recrystallization and particle stimulated recrystallization, are also discussed.

As seen in Fig. 7, grain boundaries are not smooth, in which a serrated-wavy nature of boundaries is clear and boundaries are about to bulge. In the DDRX nucleation theory, by increasing the strain upon the critical strain, grain boundaries become wavy and eventually bulge out. Grain boundary bulging creates

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new fine grains along pre-existing boundary during straining. At the peak strain, bulges are separated from primary boundaries and form new grains. A more magnified part of Fig. 7 shows a bulge that is separated from the boundary and has formed a new grain. By increasing the strain, more DRX grains form and an extensive evidence for the formation of the first layer of necklace structure (along the pre-existing grain boundaries) through the bulging mechanism is observed (Fig. 8). At the strain close to steady state, the necklace structure is completed. In addition, when the steady state of strain is realized, new grain gradually grows towards the inside of primary grains as a result of full dynamic recrystallization.

Based on hot compression flow curves (Fig. 2) and the microstructures at different strains, it can be deducted that continuous dynamic recrystallization is predominant at all deformation conditions in the present study. The



Fig. 7 SEM micrographs obtained after hot compression at 400 $^{\circ}\text{C}$ and 0.1 s^{-1} at peak strain



Fig. 8 Necklace microstructure obtained after hot deformation under strain rate of 0.1 s⁻¹ and temperature of 325 °C at peak strain

stress-strain curve of DDRX shows a clear and prominent peak due to the reduced density of dislocations caused by grain boundary migration. This will lead to the lower rate of the work hardening. Other parameters like texture, primary grain size, alloying elements, and precipitates can affect recrystallization phenomenon as well. Studies by TAN and TAN [27], SITDIKOV et al [29], GALIYEV et al [30,31] on AZ31, AZ61 and ZK60 showed the occurrence of CDRX that is due to the formation of new grains caused by misorientation of subgrain boundaries. At temperatures ranging from 200 to 350 °C, dynamic recovery is dominant, which results in creation of new grains [28]. As seen in Fig. 7, β precipitates, in white color, are present inside the grains and also grain boundaries while new grains are formed independent of the precipitates. The results of other researchers [21,25] on the other AZ series of magnesium alloys at temperatures above 350°C indicated that the occurrence of dynamic recrystallization is discontinuous. According to these studies and the present study, it can be concluded that the mechanism of recrystallization in the Mg alloys containing aluminum and zinc is mainly a function of the deformation temperature. Parameters such as texture, initial grain size and applied stress state only alter the kinetics of recrystallization and grain size while the only parameter affecting the mechanism of recrystallization is temperature.

4 Conclusions

1) At all tested temperatures, by increasing strain, the number of recrystallized grains first increases and they reaches a maximum value and decreases.

2) The fraction of recrystallized grains increases exponentially with increasing the strain and follows Avrami equation (m'=1.4).

3) Wavy/serrated grain boundary at peak strain shows inconsistent recrystallization behavior.

4) Recrystallization mechanism in AZ91 Mg alloy at the temperature ranging from 350 to 425 °C follows a necklace mechanism.

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AZ91 镁合金的热力行为和显微组织演化

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摘 要: 研究AZ91镁合金在热压缩过程中的流变行为和显微组织演化。在350~425 ℃对试样进行热压缩变形。 在应变速率为0.1 s⁻¹时,应变分别为峰应变、0.3和0.5。使用光学和扫描电子显微镜研究显微组织的演化。结果 表明,在压缩过程中再结晶晶粒沿预先存在的晶界形核;动态再结晶晶粒的数量随着应变的增大呈指数增加, 且服从Avrami方程;动态再结晶晶粒的尺寸在开始时增大,达到最大值后开始减少。 关键词: AZ91镁合金;热压缩;显微组织演化;再结晶;峰应变