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Texture evolution in AZ91 alloy after hot rolling and annealing

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Abstract: A commercial AZ91 magnesium alloy with a random initial texture was subjected to homogenization heat treatment (solution annealing) at 450 °C. This resulted in the appearance of a weak triple $(0001)-(10\bar{1}0)-(11\bar{2}0)$ fibber texture. After hot rolling at 400 °C, the deformation texture was typically (0001) basal with splitting of the basal poles in the rolling direction. After annealing at 450 °C for 36 h, the annealing texture was the retained deformation texture with more homogeneous distribution of poles around the ideal basal orientation. There was no trace of abnormal grain growth.

Key words: AZ91 alloy; texture; hot rolling; recrystallization

1 Introduction

Over the last decades, the world has had an increasing interest in the Mg-based alloys for applications in the aerospace, aircraft, and automotive industries due to their low density and high specific strength. The AZ91 (Mg−9Al−1Zn−0.2Mn, mass fraction, %) is the most favored magnesium alloy used in approximately 90% of all magnesium cast products [1]. Structural applications of Mg alloys depend on the development of plastic-forming technologies, such as hot rolling, extrusion, forging, and press forming [2]. The main obstacle to the development of such technologies is the poor workability of Mg alloys due to their crystallographic structure, and often, the presence of a basal texture [3,4]. A subsequent recrystallization of Mg alloys such as AZ31 retains this texture after full recrystallization [5,6]. The basal texture is considered inadequate for deformation because it places most of the grains in an orientation that is difficult to deform. This results in stress concentration and premature failure.

Many processing technologies of Mg alloys (e.g. equal channel angular extrusion (ECAE) [7], differential speed rolling [8], torsion extrusion [9] and continuous extrusion [10]) were effective in developing weak or non-basal textures. Recently, it has been reported that more randomized texture could be obtained through alloying additions of yttrium (Y) and rare-earth elements such as neodymium (Nd) and cerium (Ce) [11,12]. A strong on-going research activity is devoted to this goal.

Grain refinement and texture control could improve the plastic workability, as well as the strength and mechanical anisotropy at ambient temperature [13,14]. Many authors attempted to study the texture evolution during and after thermo mechanical processing of Mg-based alloys [5,6,13−15]. Most of them were dedicated to the commercial wrought AZ31 alloy. However, a study of the texture evolution of the as-received, homogenized, hot deformed and annealed AZ91 alloy is still lacking.

In this study, the texture of a commercial AZ91 magnesium alloy was studied after homogenization, hot rolling, and recrystallization treatment.

2 Experimental

The AZ91 alloy investigated was provided by Hydro Aluminium R&D Bonn in the form of rolled cast sheets with 4.5 mm in thickness. Before rolling, in order to dissolve the precipitates and ensure a solute homogenization as already shown by XU et al [16], the material was solution annealed at 450 °C for 72 h. The rolling experiment was conducted at a nominal rolling temperature of 400 °C, with the aim of avoiding precipitation of the β-phase ($Mg_{17}Al_{12}$). The total reduction in thickness was 80% with a 10% reduction per pass. The samples were heat treated at 400 °C for 10 min

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between passes to stabilize the rolling temperature. After rolling, the samples were directly quenched in water then annealed at 450 °C for 36 h. It is to be noted that at this temperature, the alloy could not undergo any massive phase transformation like continuous or discontinuous precipitation of the $Mg_{17}Al_{12}$ equilibrium phase.

Specimens for optical microscopy were prepared by conventional grinding and diamond polishing, finishing with a colloidal silica solution. Etching was done with acetic picral solution to visualize the grains and grain boundaries.

The macrotexture was determined in the mid-plane of the specimens by measuring incomplete pole figures ($5°\leq\alpha\leq75°$) in the back reflection mode using Co K_α radiation in an X-ray-texture goniometer. A set of six pole figures [$\{10\bar{1}0\}$, $\{0002\}$, $\{10\bar{1}1\}$, $\{10\bar{1}2\}$, $\{11\bar{2}0\}$, and $\{10\bar{1}3\}$] was used to calculate the orientation distribution functions (ODF) using MTEX toolbox [17]. Sample symmetry in the ODF calculation was assumed to be triclinic.

3 Results and discussion

The optical micrographs of AZ91 alloys and their pole figures are shown in Fig. 1 and Fig. 2, respectively.

The as-cast microstructure of the AZ91 alloy shown in Fig. 1(a) is mainly composed of a semi-dendritic microstructure of the δ-Mg solid solution and the β-$Mg_{17}Al_{12}$ intermetallic phase precipitated in the interdendritic spaces (dark phase).

Figure 2(a) shows the recalculated (0002), $(10\bar{1}0)$, and $(11\bar{2}0)$ poles figures of the as-received AZ91 alloy. The texture of the alloy is random, a typical case for as-cast Mg alloys with grain inoculants [18,19]. It is believed that the semi-dendritic microstructure is responsible for the absence of evident texture.

The texture of the supersaturated AZ91 alloy after homogenization and solution annealing at 450 °C for 24 h is shown in Fig. 1(c). As shown, the texture strength is obviously the same as that of the as-cast one, but it is clear that certain weak (0001), $(10\bar{1}0)$, and $(11\bar{2}0)$ fibber textures are starting to build up. Such a triple fibber texture emerging after solutioning could result from a mechanism of discontinuous dissolution that can appear in Mg-based alloy as reported in Ref. [20], but it cannot be the case presently because the original as-cast structure is of dendritic type as evidenced in Fig. 1(a).

Figure 1(b) presents the microstructure of the alloy in the as-deformed state. Non-uniform dynamically recrystallized grains (DRXed grains) visible with the un-DRXed original grains (see arrows) then produced a microstructure with a bimodal distribution of grain sizes with mean values of 5 and 25 μm, belonging to DRXed and un-DRXed, respectively. The DRXed grains exhibit

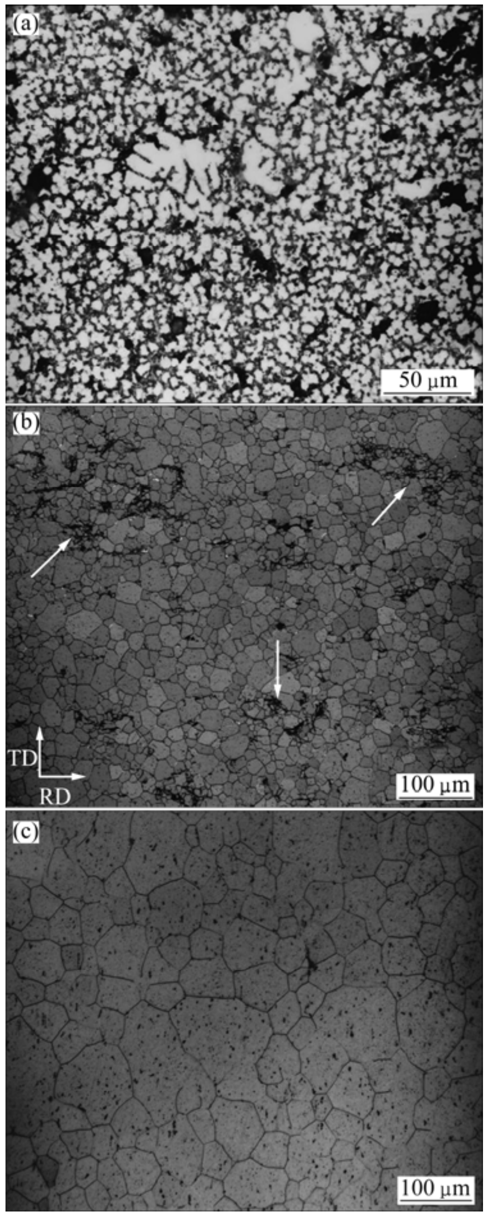

Fig. 1 Optical micrographs showing microstructure of AZ91 alloy: (a) As-cast; (b) As-rolled; (c) Annealed at 450 °C for 36 h (RD and TD stand for rolling and transverse directions, respectively)

the well-known necklace microstructure [21]. DRX has not developed considerably, leaving large zones of deformed grains. Here, twins are seldom revealed by optical micrography.

In a recent work on the same alloy hot deformed by compression at 400 °C, XU et al [16,22] observed that, contrarily to us, an almost uniform DRXed grains with few relatively coarse un-DRXed grains were obtained. This microstructure difference may be explained by the deformation process (hot rolling versus hot compression), as well as deformation conditions (temperature, strain, and strain rate). Furthermore, it is worth noting that there was a temperature drop during rolling that increased with

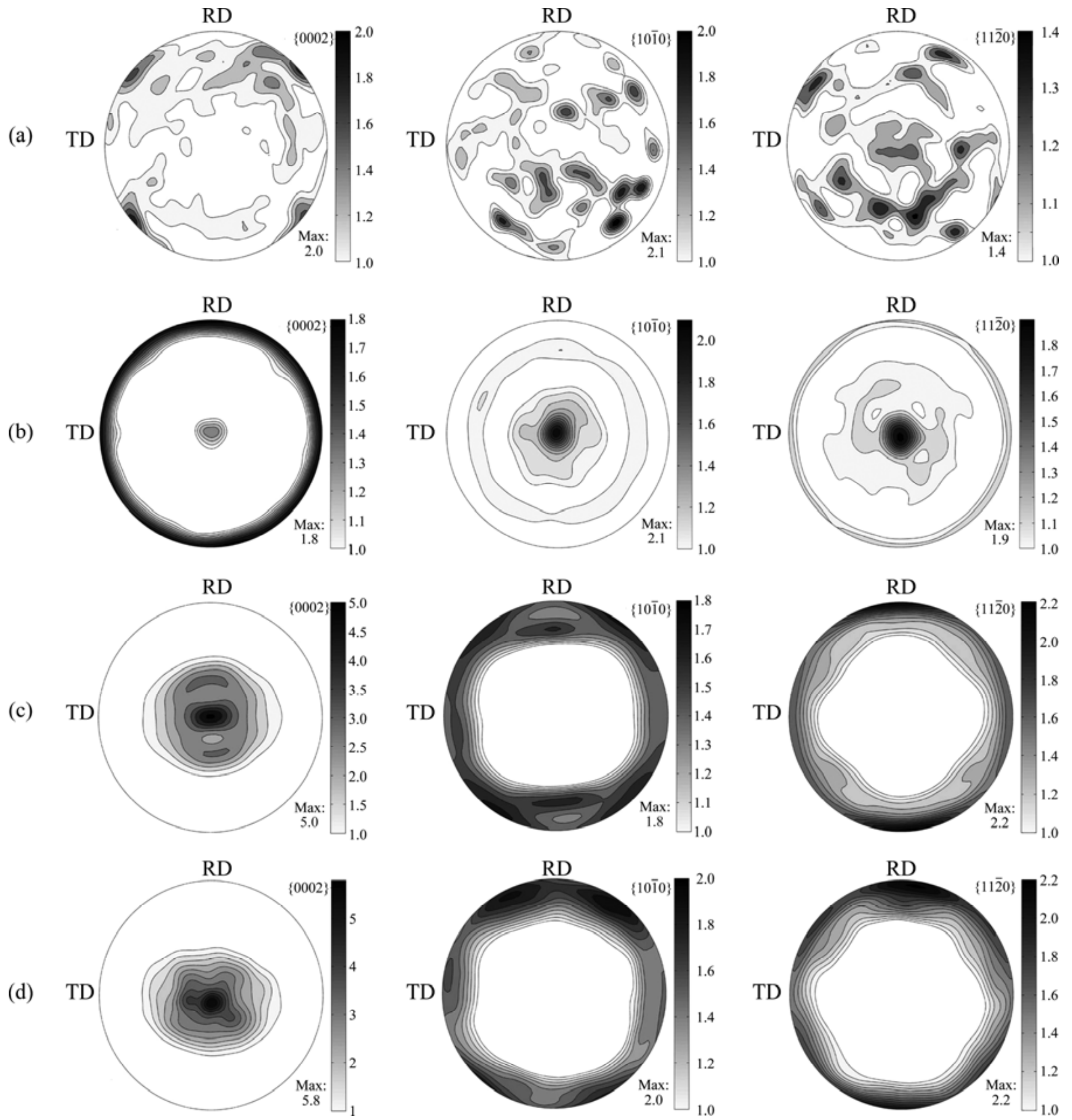

Fig. 2 (0002), $(10\bar{1}0)$ and $(11\bar{2}0)$ recalculated pole figures of AZ91 alloy: (a) As-cast; (b) After homogenization at 450 °C for 72 h; (c) As-rolled; (d) Annealed at 450 °C for 36 h

decreasing thickness, and the real hot rolling temperature was not 400 °C. This temperature drop affected the hot rolled microstructure, dynamic recovery, and recrystallization as well as precipitation stability. According to results of AL-SAMMAN et al [23], when deforming at a relatively low temperature and a high strain rate, DRX is postponed until a certain critical strain is reached.

MWEMBELA et al [24] pointed out that in Mg the level of dynamic recovery (DRV) is sufficiently low that DRX ensues above 240 °C, giving rise to new fine grains at the boundaries of the initial grain.

Figure 1(b) shows also a general thickening of the grain boundaries. This thickening is probably associated in some cases with the initiation of discontinuous precipitation, as already mentioned by BETTLES [25]. At this magnification (Fig. 1(b)), we cannot see any extended cells of discontinuous precipitation.

In our opinion, up to now, discontinuous and continuous precipitation domains of $Mg_{17}Al_{12}$ precipitation in AZ91 alloy are not definitively established like in binary Mg−Al system [26]. XU et al [16,22] studied the effect of $Mg_{17}Al_{12}$ precipitate on the microstructural changes and mechanical properties of hot compressed AZ91 alloy. They evidenced a concomitant discontinuous and continuous precipitation when deforming the alloy up to 360 °C, while at 400 °C only the second type of precipitation was present. They assumed that the $Mg_{17}Al_{12}$ precipitated phase exhibited a strong influence on the DRX behaviour, and the change in amount and morphology of the precipitates were expected to result in the change in DRXed grain size and DRX ratio even the orientation of new DRX grains.

Figure 2(c) presents the texture of supersaturated solid solution AZ91 alloy after hot rolling at 400 °C (80% thickness reduction). It is characterized by the basal planes (i.e. *c*-axis) parallel to normal direction of the sheet with a certain slight splitting of the poles in the rolling direction. This type of texture has often been observed in conventional Mg sheet alloys [5,6,15], and was considered a result of increasing the activity of pyramidal $\langle c+a \rangle$-slip and double-twinning, which causes a tilt of basal poles about ND of approximately 35°.

In the case of increased thermal activation of non-basal slip modes, the splitting of basal poles around TD is expected [27]. Moreover, the appearance of the basal texture depends strongly on the initial texture of the material [16]. A recent work of ABDESSAMEUD et al [6], devoted to studying the impact of initial texture on its evolution after uniaxial compression at 430 °C of an extruded AZ31 alloy, showed that the deformation of initial samples with compression direction parallel or normal to the extrusion direction led to a weak or strong basal texture, respectively.

The disappearance of the two $(10\bar{1}0)$ and $(11\bar{2}0)$ fibers is due to the activation of the basal slip and twinning during the deformation. The activation of twinning would cause an immediate 86° $\langle 11\bar{2}0 \rangle$ reorientation of the twinned part of grains.

The only difference of AZ91 alloy compared to AZ31 is that the maximum intensity is lower for the former. Recently, ABDESSAMEUD et al [5] have shown that the same texture evolution was evidenced in AZ31 alloy under the same deformation conditions. The maximum intensity of the basal texture was found 14 multiple of random distribution (mrd), while in the present work it is only 5 mrd. This considerable difference could be explained by the effect of $Mg_{17}Al_{12}$ precipitates. We can speculate then that new DRX grains may exhibit orientations that can be far from basal texture, weakening the overall texture intensity.

Very recently, LI et al [28] have addressed the role of $Mg_{17}Al_{12}$ equilibrium precipitate in the deformation mechanisms and the texture for AZ Mg alloys. They found that this phase reduced twinning activity, and then enhanced DRX, leading to weak deformation textures at high strains. While at 400 °C and 10^{-4} s^{-1}, the dissolution of the $Mg_{17}Al_{12}$ equilibrium phase resulted in a sharp texture.

Many studies [29−31] have addressed the texture evolution of Mg-based alloys; they found that the texture of the DRX grains followed that of the parent grains closely in all alloys, implying that the DRX texture is dominated by the deformation conditions, rather than by the preferred nucleation or growth.

Figure 1(c) presents the microstructure of hot deformed AZ91 alloy annealed at 450 °C for 36 h. The annealing treatment resulted in a fully recrystallized microstructure, and in normal grain growth with an average grain size of 56 μm. Normal grain growth has been already evidenced in hot rolled AZ31 alloy (d ~30 μm for 10^{-2} s^{-1} and 450 °C) [6], and WE54 hot deformed by uniaxial and plane strain compression (d ~20 μm for 10^{-2} s^{-1} and 400 °C) [11]. Our results are in agreement with those of AL-SAMMAN et al [32], who have assumed that, contrary to pure Mg and some other binary Mg alloys, AZ91 did not undergo considerable grain growth. This must be caused by a strong effect of solutes (Zener Drag) or precipitates that are more present in this alloy than AZ31 alloy.

The recrystallization−annealing texture of the AZ91 alloy is shown in Fig. 2(d) through recalculated {0002}, $\{10\bar{1}0\}$, and $\{11\bar{2}0\}$ poles figures. It can be seen that it is almost the same as the deformation texture, i.e., retained basal texture with no other texture components. Similar observations were made for AZ31 alloy [5,13]. These authors have shown that such a stable basal texture existed even after full annealing at high temperature without emerging or strengthening any minor components. It should be noted that the splitting of basal poles has been replaced by a homogeneous distribution around the ideal basal orientation. Such a texture has already been observed in AZ31 alloy hot rolled at 400 °C after 80% of thickness reduction, and annealed at 450 °C for 10 d [8]. Unlike the cubic materials, the annealing texture in Mg and its alloys is often close to the deformation one [5,6,13], and is interpreted as continuous recrystallization or strong recovery. It has been reported that static annealing after deformation leads to only small texture changes [33,34].

Of particular interest is the intensity of the basal poles for different state of AZ91 alloy as a function of tilt angle towards TD (Fig. 3). As pointed out by ION et al [29], this is a more convenient presentation of the texture and depicts the most important features of pole figures. In the case of as-rolled and recrystallized AZ91 alloy, a sudden drop of texture intensity was observed within 0−20° about ND (typical basal texture). By contrast, the basal pole intensity of as-cast AZ91 alloy dropped randomly along the tilt angles. With respect to tilt towards TD, the intensity distribution of homogenous AZ91 alloy increased between 80 and 90°, which is attributable to the $(10\bar{1}0)$ and $(11\bar{2}0)$ fibbers.

Table 1 summarizes the quantitative texture parameters determined by the MTEX software, which are: the maximum basal pole intensity, volume fraction of the basal fiber, and the texture index given by [35]

$$I = \frac{1}{8\pi^2}\int_G |f(g)^2|\,\mathrm{d}g \tag{1}$$

where $f(g)$ is the ODF value and G is the Euler space.

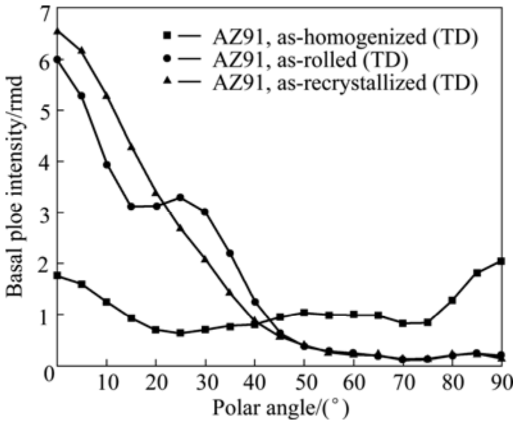

Fig. 3 Basal pole intensity distribution for different states of AZ91 alloy as function of tilt towards TD

Table 1 Quantitative texture parameters determined by MTEX software for different states of AZ91 alloy

State	(0002) pole figure intensity/mrd	F_v of basal fiber	Texture index
As-received	1.9	2.22	1.19
Homogenized	1.8	3.64	1.12
Hot rolled	5.1	12.66	2.02
Annealed	5.8	14.19	2.16

There is a substantial increase of the parameters from the as-received/homogenized to hot rolled/ recrystallized alloys, and these parameters are very close for the as-received and homogenized alloy, but a very slight increase for the annealed and rolled alloy.

PÉREZ-PRADO and RUANO [33] discussed the case where a single strong texture is present in the microstructure with a large number of lower-angle boundaries in a random distribution of grains. They assumed that relative growth of the grains depends strongly on the grain boundary character distribution, with a certain privilege for grains with high angle grain boundaries. Very recently, some of the present authors have studied the grain boundary character distribution effect on the discontinuous precipitation in AZ91 alloy [36]. Figure 4 shows the histogram of the grain boundary misorientation, corresponding to the supersaturated solid solution AZ91 after hot rolling and annealing at 450 °C for 36 h. The random distribution of grain boundaries is given in full line. In agreement with Ref. [37], the distribution revealed common special boundaries, with the maximum densities near 30° and 86°−90°, as well as in the absence of other orientation relationships with perceivable densities that could be indicative of new special boundaries. Following Refs. [22,38], these peaks correspond to $\{10\bar{1}1\}$ double twin, and $\{10\bar{1}2\}$ primary tensile twin, respectively. The misorientation distribution peak at 30° is very common in hexagonal materials, but the mechanisms behind it are not always the same. In the current case, one could expect that it is attributed to basal poles rotation about the *c*-axis during static recrystallization after rolling.

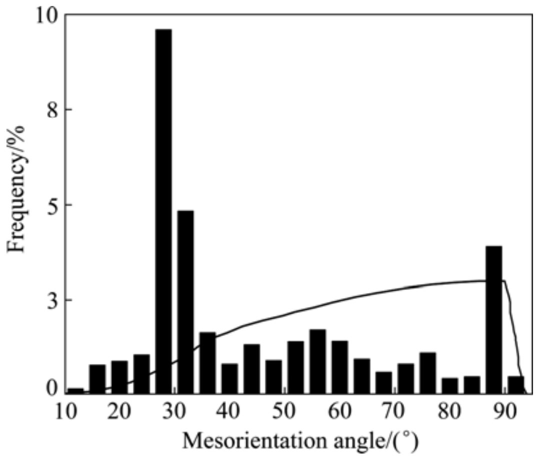

Fig. 4 Histogram of measured misorientation angle distribution for AZ91 alloy hot rolled and recrystallized at 450 °C for 36 h (The line represents the density function for a random misorientation distribution)

As a result, neither the volume fraction of the basal fibber is strong nor the frequency of low angle grain boundaries is high. These observations allow us to conclude that the speculations in Ref. [33] may not be valid in our present study. A special thermo- mechanical processing and/or rare earth (RE) elements alloying (weak non-basal textures are often associated with complex mischmetal additions, rather than a single alloying element) [11,39] is necessary in order to promote randomized texture in the present alloy. This is the subject of ongoing research activity.

4 Conclusions

1) The as-received commercial AZ91 magnesium exhibited a random initial texture.

2) Homogenization heat treatment at 450 °C for 72 h resulted in the appearance of triple fiber texture basal, $(10\bar{1}0)$, and $(11\bar{2}0)$.

3) After hot rolling at 400 °C, deformation texture was typically basal with splitting of the basal poles in the rolling direction.

4) Annealing texture after annealing at 450 °C for 36 h was the same and retained basal deformation texture with a homogeneous distribution of basal poles around the ideal basal orientation. Annealing also resulted in normal grain growth.

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AZ91 镁合金热轧退火过程中的织构演变

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摘　要：将具有随机初始织构的工业 AZ91 镁合金在 450 °C 进行均匀化热处理。经热处理后，合金中出现弱的 $\{0001\}-\{10\bar{1}0\}-\{11\bar{2}0\}$ 三重纤维织构。经 400 °C 热轧后，合金的变形织构是典型的(0001)基面，其基极沿轧制方向分裂。在 450 °C 退火 36 h 后，其残余变形织构中的基极沿基面分布更均匀，没有出现晶粒的异常长大。

关键词：AZ91 镁合金；织构；热轧；再结晶

(Edited by Hua YANG)