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ARTICLE

Evolution of Microstructure in Al-Mn Alloy with a Low Ratio of Fe/Si During Homogenization

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Abstract: The evolution of microstructure in Al-Mn alloy with a low ratio of Fe/Si produced by twin-roll casting (TRC) during various homogenization treatments was investigated. The grain structure near the surface transforms from fibrous to coarse elongated recrystallized grain structure during homogenization at 550 °C for 4 h. The grain in the interior tends to be equiaxed with the temperature increasing. The primary particles are broken up after homogenization at 450 and 500 °C for 4 h, and coarsened when the temperature increases to 550 °C. The evolution of dispersoids is controlled by nucleation and growth mechanisms at 450 and 500 °C for 4 h, whereas the coarsening and dissolution are predominant mechanisms during homogenization at 550 °C for 4 h. Many small Zr-bearing dispersoids are obtained during homogenization at 500 °C for 4 h.

Key words: Al-Mn alloy; homogenization; microstructure; primary particle; dispersoids

Al-Mn alloy is widely used as fins in automotive heat exchangers, due to its high thermal conductivity, good corrosion resistance and good formability as well as low cost[1,2]. The fins are usually produced with thickness below 0.1 mm. Thus, in order to avoid collapse during its application, maintenance of good sagging resistance and recrystallization resistance is demanded. The addition of Zr, which can lead to formation of a coherent Al_3Zr phase, has been proposed to impede recrystallization in aluminum alloys[3,4].

The effect of alloying elements on microstructure and properties in Al-Mn alloys has been extensively investigated. Orthorhombic Al_6(Mn,Fe) and cubic α-Al(Mn, Fe)Si are the main Mn-bearing particles in Al-Mn alloy, while Fe and Si remarkably promote the precipitation of them[5]. In low Si content Al-Mn alloy, Al_6(Mn,Fe) are the main second phase particles, while the volume fraction of α-Al(Mn,Fe)Si would be prevailing with the increase of Si content[6]. Besides, high Si content alloy has a larger number of fine particles than high Fe content alloy. Moreover, both Fe and Si elements are able to facilitate the precipitation of Al_3Zr particles, and Si can stabilize the metastable Al_3Zr phase[7,8].

Higher solid solution supersaturation and smaller grain size would be achieved owing to high solidification rate in twin-roll casting[9,10]. Hence the TRC Al-Mn alloys have a greater potential of precipitation during homogenization and downstream heat treatment, compared to direct-chill casting alloys. The size, quantity and distribution of intermetallic constituent particles and dispersoids have a strong effect on the microstructure evolution and the recrystallization kinetics of the Al-Mn alloys. It is established that fine particles can exert a drag force (Zener drag) on both dislocations and boundaries which will inhibit the recrystallization process. Coarse particles (>1.0 μm), usually formed during casting, may act as nucleation sites to promote recrystallization by particle stimulation nucleation (PSN) mechanism[11,12]. Studies on evolution of particles, including constituent particles and dispersoids, during homogenization in Al-Mn alloy with a high ratio of Fe/Si (Fe/Si>1) have been numerous[13,14]. However, investigations on evolution of particles in Al-Mn alloy with

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a low ratio of Fe/Si have been scarce. In the present work, the microstructural evolution and quantitative analysis of primary particles and dispersoids in Zr-containing Al-Mn alloy with a low ratio of Fe/Si during homogenization have been investigated. The interaction between primary particles and dispersoids has been discussed as well.

1 Experiment

The material used in this experimental work was a twin-roll cast strip in industrial conditions, with 7.0 mm in thickness. The chemical composition (wt%) of the alloy was: Mn 1.5, Fe 0.3, Si 0.6, Zn 1.0, Zr 0.1. Three different homogenization treatments were conducted in an air circulation furnace with a temperature accuracy of ±2 K. Samples were heated to 450, 500, 550 °C with a heating rate of 40 K/h and held for 4 h at the target temperature followed by immediate quenching into cold water. Both Vickers hardness and electrical conductivity (σ) were measured on the RD-TD plane at room temperature (about 293 K). The morphology of primary particles and dispersoids were performed by scanning electron microscope (SEM) and transmission electron microscope (TEM). The composition of particles was investigated by energy dispersive spectroscopy (EDS) attached to SEM and TEM. Characteristic size parameters of particles were obtained by IMAGE J software. TEM foils were prepared through electrolytic polishing in a solution of 33% nitric acid in methanol at −20 °C. Foils were observed in a JEOL JEM-2100F TEM at 200 kV. The grain structure was measured in polarized light after anodizing with Barker's reagent. For all the micrographs measured by optical microscopy (OM) and SEM observation, the horizontal and vertical directions correspond to rolling and normal directions (RD-ND), respectively.

2 Results and Discussion

2.1 Microhardness, electrical conductivity and grain structure during homogenization

The microhardness of the samples during different homogenization treatments is shown in Fig. 1. The reduction rate increases with the temperature increasing. The microhardness decreases in an important manner when the sample is heated to 550 °C.

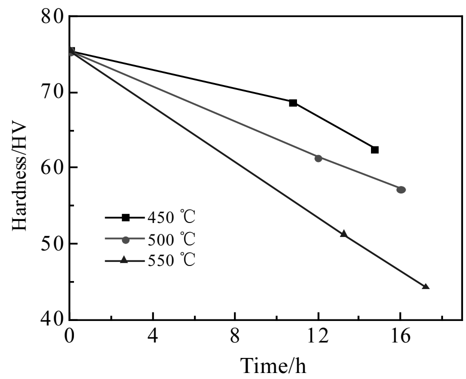

Fig. 1 Microhardness of the samples during different homogenization treatment

The shape of grain in the interior of strip shows a negligible change during different homogenization treatments. The aspect ratios of grain in the interior at as-cast state, 450 °C/4 h, 500 °C/4 h and 550 °C/4 h are 11.16, 9.02, 7.44 and 6.34, respectively. It is indicated that the grain structure tends to be equiaxed with the increase of temperature. The grain structures near the surface of the samples during different homogenization treatments are shown in Fig. 2. The as-cast alloy has a typical fibrous grain structure, and the samples homogenized at 450 and 500 °C for 4 h still maintain a deformed grain structure with a very few elongated recrystallized grains. However, a large number of coarse elongated recrystallized grains along RD direction are obtained when temperature increases to 550 °C, which are attributed to the formation of fine particles near the surface of strip. It is established that the supersaturation of the surface is high owing to high solidification rate in twin-roll casting. These fine and dense particles distributed along RD direction would offer a more significant pinning effect on moving sub-grain/grain boundaries along ND direction than the effect on boundaries along RD direction, which finally leads to the formation of coarse elongated recrystallized grains along RD direction.

The relationship between σ and the concentrations of solutes in solid solution can be expressed as[15]:

$$\sigma = 0.0267 + 0.032\mathrm{Fe_{ss}}\% + 0.033\mathrm{Mn_{ss}}\% + 0.0068\mathrm{Si_{ss}}\% + 0.0021\mathrm{Particles}\% \quad (1)$$

where Fe_{ss}%, Mn_{ss}% and Si_{ss}% are the mass fractions of these elements in solid solution. It can be seen that Si_{ss} has a negligible influence on σ, while all Fe forms intermetallic particles during casting. Therefore, the evolution of σ can be used to monitor the concentration change of Mn in solid solution and reflect the degree of decomposition of the supersaturation solid solution during homogenization.

The evolution of σ during homogenization is plotted in Fig. 3. When the samples are homogenized at 450 and 500 °C for 4 h, all of three σ increase quickly with heating, and then increase slightly with the holding time, while the σ starts to decrease with prolonging holding time at 550 °C. The value of σ reaches a maximum at 500 °C for 4 h. It is indicated that the supersaturation solid solution obtained by twin-roll casting initiates to decompose and Mn solutes start to precipitate from matrix with the increase of temperature. However, when homogenization temperature reaches to 550 °C, Mn solutes begin to dissolve into matrix.

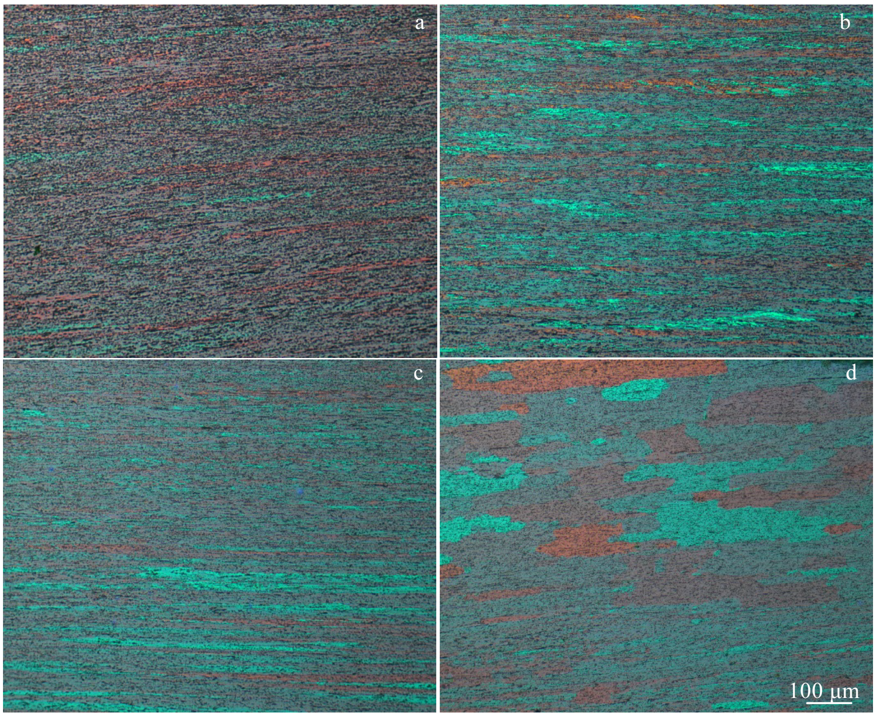

Fig. 2 OM images in the strips surface of samples during different homogenization treatments: (a) as-cast state, (b) 450 °C/4 h, (c) 500 °C/4 h, and (d) 550 °C/4 h

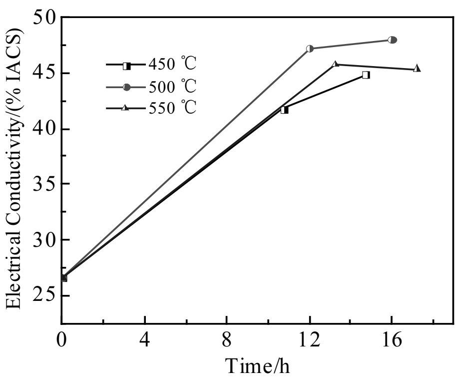

Fig. 3 Electrical conductivity of the samples during different homogenization treatment

2.2 Primary particles analysis during homogenization

The evolution of size and area fraction in as-cast state and variation homogenization are exhibited in Table 1.

A large number of rod like and lamellar eutectic primary particles distributed along with RD direction are observed in as-cast state (Fig. 4a). Most of them have been determined to be α(Al)+α-Al(Mn, Fe)Si eutectic structure and a mixture of primary Si and α(Al)+α-Al(Mn,Fe)Si, and no Al_6(Mn,Fe) eutectic phase has been observed, which is consistent with Karlík's research that Al_6(Mn,Fe) phase is barely formed in Al-Mn alloy with a low ratio of Fe/Si[16]. The lamellar eutectic structure is broken up into small fragments after homogenized at 450 °C for 4 h (Fig. 4b). The lamellar eutectic structure in as-cast state is further broken up into the chain structure when the temperature reaches to 500 °C, as shown in Fig. 4c. Nevertheless, Fig. 4d shows that the chain structure of particles is coarsened during homogenized at 550 °C for 4 h.

The size of primary particles decreases with increasing temperature and achieves a minimum at 500 °C for 4 h, whereas it tends to increase when temperature reaches to 550 °C. The evolution of area fraction shows the same tendency as the size. It is suggested that the morphology and size of primary particles have a slight change at 450 °C for 4 h compared with as-cast sample, due to the low diffusion rate of Mn in Al matrix at low temperature. When the sample is heated to 500 °C, the efficient dissolution of primary particles is obtained, owing to the acceleration of solutes diffusion rate with the increase of temperature, whereas primary particles tend to coarsen when temperature increases to 550 °C.

Table 1 Diameter, area fraction of primary particles in as-cast, the samples during different homogenization conditions

Sample state	Diameter/μm	Area fraction/%
As-cast	1.502	4.275
450 °C/4 h	1.373	3.930
500 °C/4 h	1.136	2.952
550 °C/4 h	1.256	3.146

2.3 Dispersoids analysis during homogenization

The morphology and distribution of dispersoids in the samples during different homogenization is shown in Fig. 5. A few small dispersoids located on dislocation is found

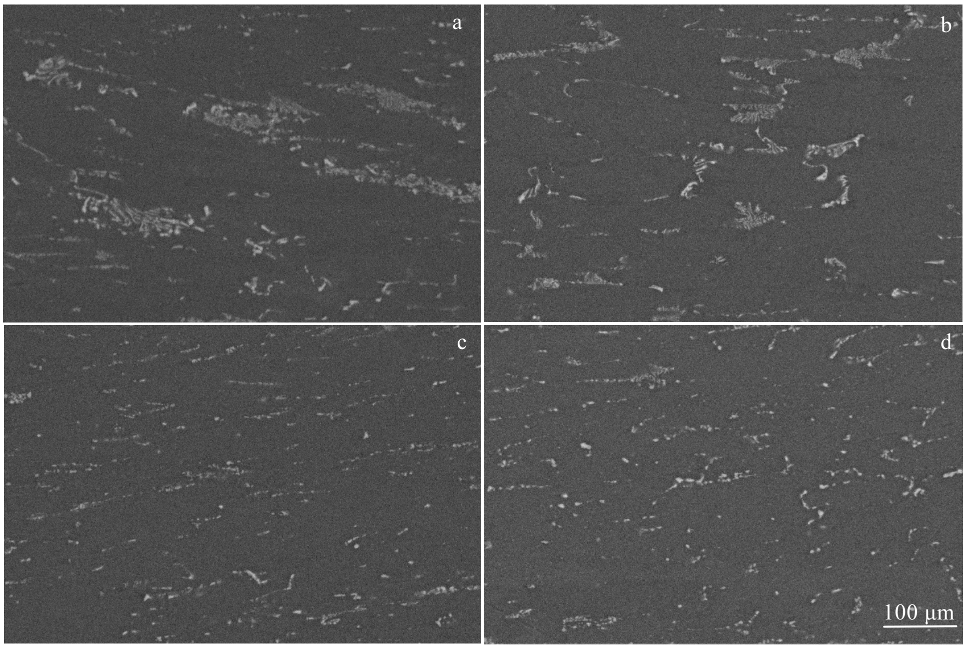

Fig. 4 SEM images of primary particles in the samples during different homogenization treatments: (a) as-cast state, (b) 450 °C/4 h, (c) 500 °C/4 h, and (d) 550 °C/4 h

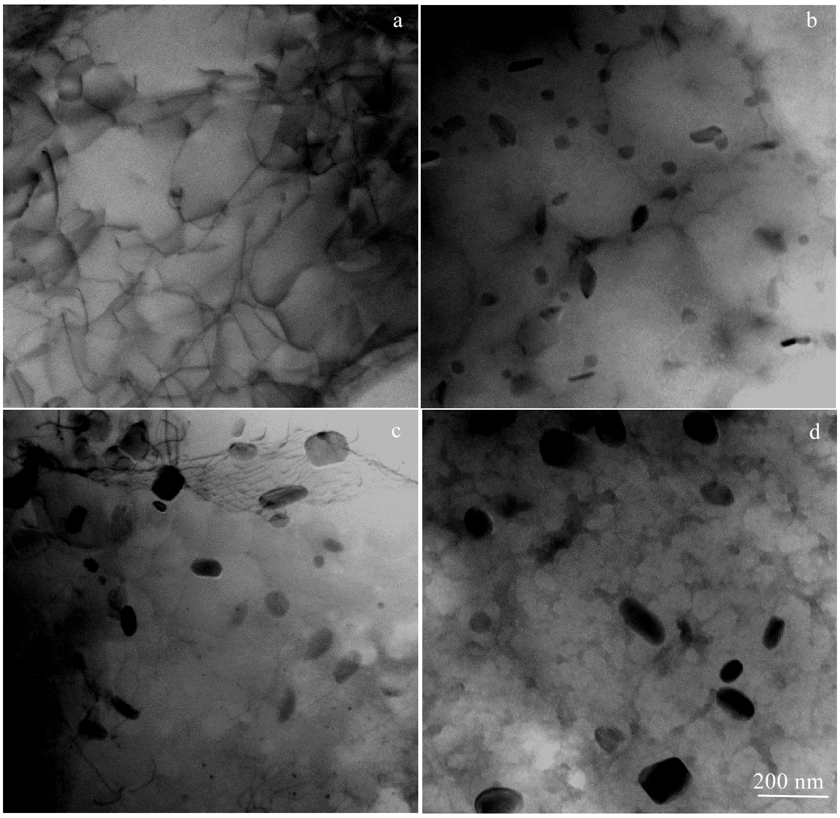

Fig. 5 TEM images of dispersoids in the samples during different homogenization treatments: (a) as-cast state, (b) 450 °C/4 h, (c) 500 °C/4 h, and (d) 550 °C /4 h

sporadically in as-cast state. Large amounts of fine rod like, plate like and equiaxed like dispersoids precipitated on sub-grain boundaries and in grains are obtained after homogenized at 400 °C for 4 h. Most of them have been determined to be α-Al(Mn, Fe)Si phase with the equivalent diameter nearly 50 nm. When the sample is homogenized at 500 °C for 4 h, α-Al(Mn, Fe)Si dispersoids have a slightly large size, compared with the sample homogenized at 400 °C, while a number of small spherical dispersoids with size less than 30 nm have been observed on dislocations, determined to be Zr-containing particles. The apparent coarsening of dispersoids has occurred after homogenized at 550 °C for 4 h. Mn-bearing dispersoids in Al-Mn alloy have a precipitation temperature range of 300~500 °C [16], and there is a limitation of precipitation behavior at low temperature due to poor performance of diffusion rate of Mn in Al matrix. The kinetics of precipitation increases with elevated temperature. The dispersoids size, area fraction evolution and size distribution in the samples during homogenization treatments are presented in Fig. 6.

The area fraction of dispersoids obtains a maximum value at 450 °C, and then it drops with the temperature. The equivalent diameter of dispersoids increases with elevated temperature, except that there is an abnormal point at 500 °C, as shown in Fig.6a. It is attributed to the precipitation of small Zr-containing dispersoids at 500 °C.

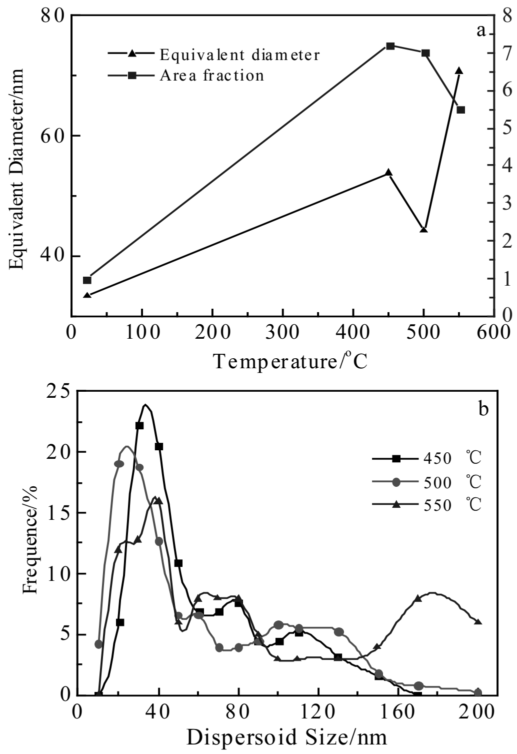

Fig.6 Equivalent diameter, area fraction evolution (a) and dispersoids size distribution (b) of dispersoids in the samples during different homogenization

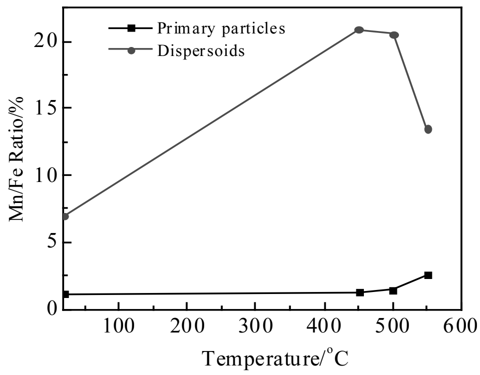

Fig.7 Mn/Fe ratio of primary particles and Mn-bearing dispersoids in the samples during different homogenization

According to the size distribution of dispersoids (Fig.6b), the sample homogenized at 500 °C for 4 h has more fine dispersoids with diameter 20~30 nm, than the sample homogenized at 450 °C for 4 h which has a main peak appearing at 30~50 nm. Al_3Zr phase has an appropriate precipitation temperature range of 400~500 °C, and a ramp heating used in heat treatment benefits to increase its number density[17]. When homogenized at 550 °C for 4 h, not only the main peak of distribution moves to right side, but a minor peak appears at large size side (>160 nm). It is indicated that the nucleation and growth are dominant mechanisms to control the evolution of dispersoids after homogenization at 450 and 500 °C for 4 h, whereas ripening and dissolution take over gradually when heating temperature over is 500 °C.

The Mn/Fe ratios of primary particles and Mn-bearing dispersoids in the samples during homogenization treatments measured by EDS are plotted in Fig. 7.

The Mn/Fe ratio of Mn-bearing dispersoids slightly decreases when temperature is elevated to 500 °C, and then it drops quickly as heating up to 550 °C. Whereas the Mn/Fe ratio of primary particles has an opposite tendency with the temperature. It is suggested that some Mn elements diffuse from Mn-bearing dispersoids into primary particles during homogenization; moreover, the diffusion tendency is accelerated at high temperature.

3 Conclusions

1) For TRC Al-Mn alloy with a low ratio of Fe/Si, fibrous structure near the surface and elongated grains in the interior are obtained in as-cast state. The elongated grains in the interior tend to be equiaxed and a few elongated recrystallized grains appear near the surface after homogenization at 450 and 500 °C for 4 h. However, extremely coarse elongated recrystallized grains occur near the surface of strip during homogenization at 550 °C for 4 h.

2) The primary particles in TRC Al-Mn alloy with a low ratio of Fe/Si are determined to be α(Al) +α-Al(Mn, Fe)Si

eutectic structure, a mixture of primary Si and α(Al)+ α-Al(MnFe)Si. The primary particles are broken up after homogenization at 450 and 500 °C for 4 h, while they are coarsened when temperature increases to 550 °C.

3) Most of dispersoids precipitated during various homogenization treatments are α-Al(Mn, Fe)Si phase. The evolution of dispersoids is controlled by nucleation and growth mechanisms at 450 and 500 °C for 4 h, and the precipitation kinetics increases with the temperature. The coarsening and dissolution are predominant mechanisms during homogenization at 550 °C for 4 h.

4) An interaction occurs between primary particles and Mn-bearing dispersoids that some Mn elements diffuse from Mn-bearing dispersoids into primary particles during homogenization. Moreover, the diffusion tendency is accelerated at high temperature.

5) Numerous Zr-bearing dispersoids have been precipitated during homogenization at 500 °C for 4 h, compared to the samples homogenized at 450 and 550 °C for 4 h.

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均匀化退火对低 Fe/Si 比 Al-Mn 合金微观组织演变的影响

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摘　要：主要研究了双辊轧制的低 Fe/Si 比 Al-Mn 合金在不同均匀化处理过程中的微观组织演变情况。当对合金进行 550 ℃/4 h 的均匀化处理时，合金表面的晶粒组织从典型的纤维组织转变成粗大的长条状再结晶晶粒组织。合金心部的晶粒组织则随着均匀化温度的升高而趋向于等轴化。当对合金进行 450 ℃/4 h 和 500 ℃/4 h 的均匀化处理时，粗大的初生相开始破碎，当均匀化温度升高至 550 ℃时，破碎的初生相发生粗化。在 450 ℃/4 h 和 500 ℃/4 h 的均匀化处理条件下，弥散相在均匀化过程中的演变主要受形核和长大机制控制，而在 550 ℃/4 h 的均匀化条件下，弥散相的粗化和回溶成为主要机制。当对合金进行 500 ℃/4 h 处理时，大量细小的含 Zr 弥散相从过饱和固溶体中析出。

关键词：Al-Mn合金；均匀化退火；微观组织；初生相；弥散相

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