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Textural inspection of recrystallization process under bimodal particle distribution in pure Al-Mn alloy①

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Abstract: Recrystallization in a pure Al-1.3% Mn alloy containing large undeformable particles was investigated at low annealing temperature with concurrent precipitation and emphasis was put on its orientational aspect. Results revealed significant changes in microstructure and microtexture due to an inhomogeneous precipitation as compared with those at high annealing temperatures. Despite the easy nucleation at large particles, precipitation substantially suppressed the growth of grains formed there and the texture was mainly influenced by the grains formed at grain boundaries.

Key words: recrystallization; precipitation; Al-Mn alloys **Document code**: A

1 INTRODUCTION

Annealing a deformed material containing large particles will lead to, due to the large misorientation and the high stored energy, preferred nucleation around particles, the so called particle stimulated nucleation (PSN). The microstructure and the texture related with PSN may be rather complicated since, firstly, PSN is influenced by the size, distribution and volume fraction of particles and by annealing temperatures, and secondly, not only PSN but also the nucleation at grain boundaries can take place. Whereas annealing at high temperature often gives rise to a fine grained structure and a nearly random texture, annealing at low temperature is often accompanied by precipitation (the so called bimodal particle distribution[1~3] in which large primary particles facilitate nucleation and small secondary particles inhibit recrystallization) and yields substantially different distribution of grain sizes[1~5]. A recent review on the subject is given in Ref. [5]. It was reported[4,6] that PSN will be more strongly suppressed by concurrent precipitation than the nucleation at other sites. Despite some inspections into the bimodal particle distribution[1~6], the proposed models for quantitative description of grain size distribution are still far from satisfactory because of the complication. Moreover, the microtextural information is still less obtained.

This work aims to investigate the recrystallization under bimodal particle distribution by revealing its orientational aspect and comparing it with that obtained at high annealing temperature[7].

2 EXPERIMENTAL

After solution treatment at 630 ℃ a pure Al-1.3% Mn(mass fraction) alloy was annealed at 400 ℃ for 170 h and at 628 ℃ for 24 h and cooled in air. The sample containing large particles was still supersaturated. The particles (Al_6Mn) possessed an average size of ~3 μm and a volume fraction of ~3.6%. The initial grain

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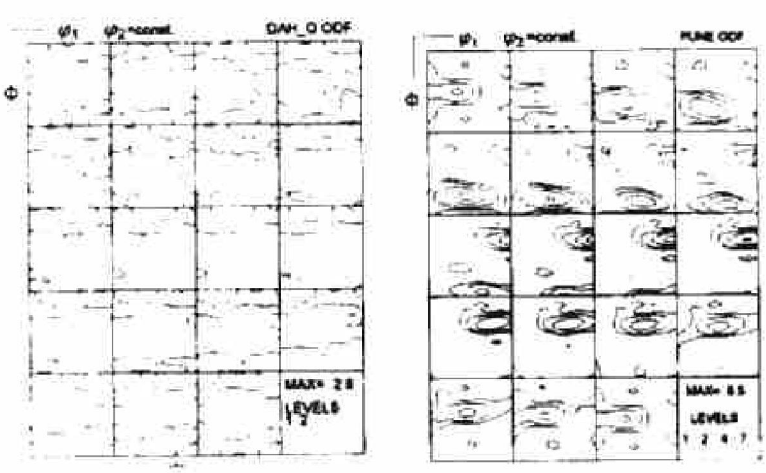
size of sample was 120 μm. This sample was then exposed to a cold rolling of 82% to 97% reduction and annealed in salt bath. Structural observation indicated that precipitation occurred in a wide range of temperatures. Above 350 ℃ precipitation occurred after the completion of recrystallization and between 300 ℃ and 350 ℃ precipitation and recrystallization happened concurrently. Macrotextures were measured by X-ray diffraction and microtextures were determined by EBSD (electron back scattering diffraction) analysis in SEM[8].

3 RESULTS AND ANALYSIS

3.1 Macrotexture

Fig. 1 shows the macrotextures of the 82% rolled samples annealed at different temperatures. At 450 ℃ no precipitation took place and, besides the weak cube texture {001}⟨100⟩ originating from the nucleation at cube bands[9], nearly random texture with weak cube_{ND} {001}⟨310⟩ (Fig. 1(a)) was obtained, which indicates the strong effect of PSN; at 330 ℃, due to the strong precipitation and consequently the occurrence of continuous recrystallization, the texture consists mainly of the retained rolling texture components. The weak peaks of cube_{RD} {013}⟨100⟩ and cube_{TD} {013}⟨031⟩ (Fig. 1(b)) were produced by discontinuous recrystallization.



(a) (b)

Fig. 1 Macrotextures of 82% rolled samples annealed at different temperatures for different times
(a)—450 ℃, 15 s; (b)—330 ℃, 3 d

From macrotextures, however, one can not clearly know whether this change in textures was related with PSN or the nucleation at other sites and this should be solved by local orientation measurements.

3.2 Microstructure

Fig. 2 shows the typical microstructure of an 82% rolled, 330 ℃, 3 d annealed sample. From this micrograph the following structural features can be found.

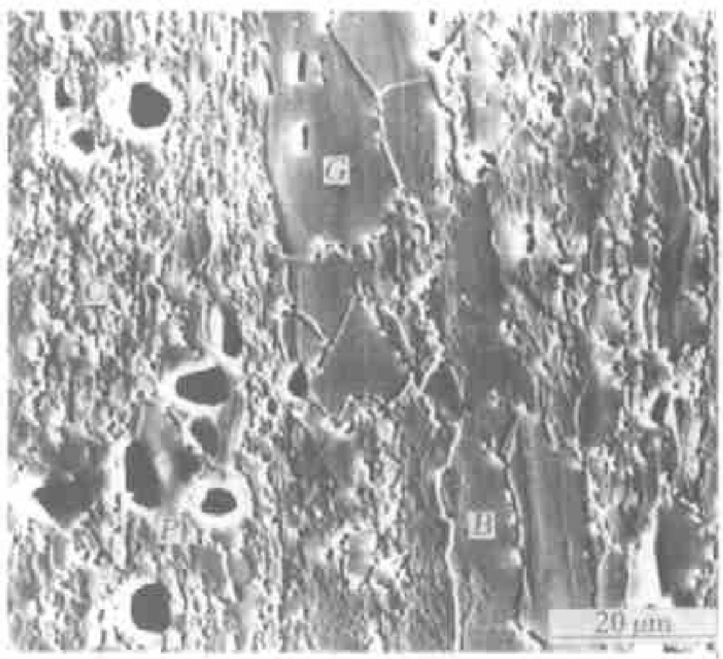


Fig. 2 Microstructure of 82% rolled, 330 ℃, 3 d annealed sample showing bimodal particle distribution

(1) Nucleation Not only PSN but also the nucleation at other sites occurred. Even at the temperature, where almost only continuous recrystallization took place (e.g. 300 ℃), the nucleation at grain boundaries was still active. This indicates that precipitation has not suppressed entirely the nucleation of a specific type.

(2) Precipitation process Precipitation totally suppressed PSN at some particles (Fig. 2, position *A*). Moreover, precipitation was quite non-uniform. There are a lot of precipitates in some deformed bands (Fig. 2, position *C*), whereas in other bands there are few precipitates (Fig. 2, position *B*). Such distribution difference of precipitates would certainly lead to a difference in grain growth. Orientation scanning by

EBSD revealed more precipitates mainly in the S {123} ⟨634⟩/C {112} ⟨111⟩ oriented matrices and less precipitates mainly in the B{110} ⟨112⟩ matrix[10]. This non-uniform distribution of precipitates was relieved by the increase of rolling degree and the decrease of initial grain size.

(3) Growth It is this distribution difference of precipitates that leads to inhomogeneous grain sizes. At the large particles located in the matrix with strong precipitation, the growth of PSN grains would be remarkably restricted. On the contrary, a slower precipitation like in region *B* of Fig.2 would provide an opportunity for the new grains to grow, referring to the small PSN grains in region *P* and the large grains at grain boundaries in region *G* of Fig.2. In contrast to Ref.[1 ~ 6] where new grains could overcome the pinning of fine precipitates, the new grains at this temperature can only grow into the regions where no precipitation occurred. Statistically, this difference in grain size is shown in Fig.3 together with the distribution of the grain sizes at 450 ℃ as a comparison. The grain sizes of the sample annealed at 330 ℃ ranged from 3 μm to 45 μm, while those of the 450 ℃ annealed sample only ranged from 3 μm to 24 μm. The average grain size at 330 ℃ (13.7 μm) is not significantly larger than that (11.4 μm) at 450 ℃. As rolling reduction increased to 97%, the difference in grain sizes between PSN grains and the grains formed at grain boundaries became larger. It is further noticed that the growth of grains enhanced the precipitation of either round or elongated precipitates at boundaries between

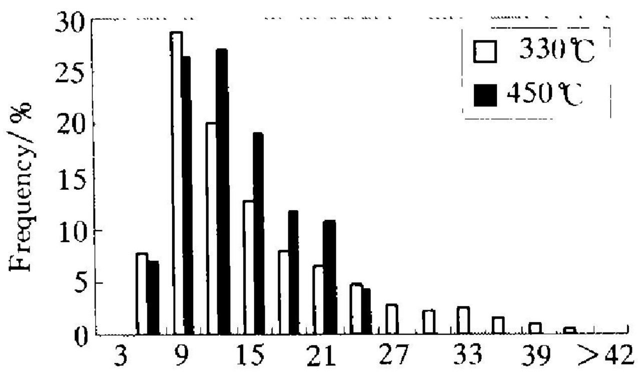


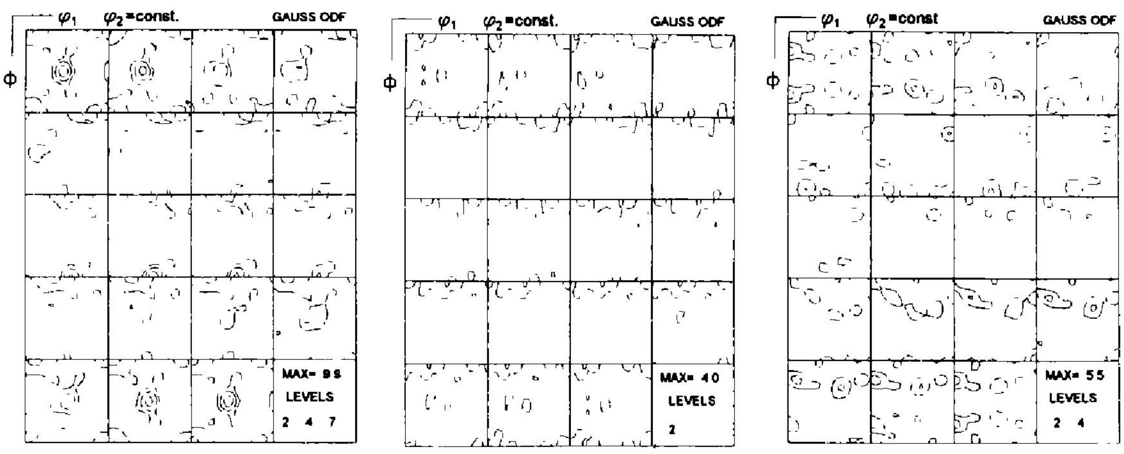
Fig.3 Distribution of grain size at different temperatures, 82% rolled samples

recrystallized and deformed grains and this may happen earlier than the precipitation in the deformed matrix.

3.3 Microtextures

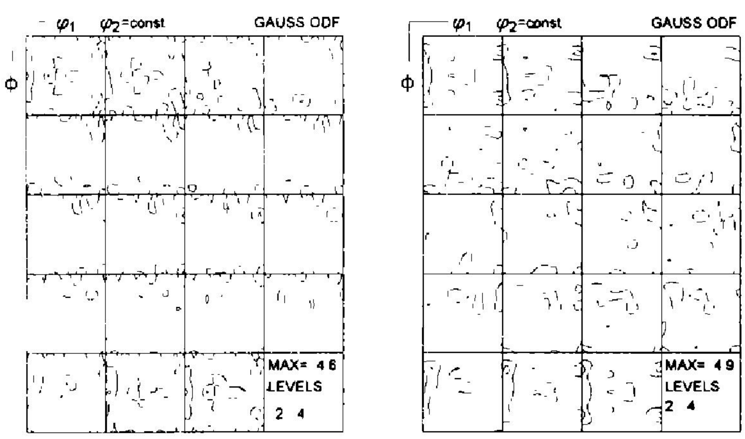
Fig.4 illustrates the microtexture evolution of PSN grains in the 82% rolled, 330 ℃ annealed specimens. At the early stages (Fig. 4 (a)) the orientation distribution shows the characteristics of PSN grains in polycrystals, namely, typical orientations of $cube_{ND}$ within deformed S/C matrices and the B and $cube_{RD}$ orientations around particles at grain boundaries[7]. The orientations of PSN grains at the early stages of recrystallization were also measured on the 97% rolled, 325 ℃, 40 s annealed sample and the 92% rolled, 300 ℃, 16 h annealed sample. Despite the changes in the maximum intensity in different samples, the $cube_{ND}$, B and $cube_{RD}$ components always appeared. This is due to the fixed orientation spectrums in deformation zones around particles and the preferential nucleation at them. Such strongly scattered subgrains were not influenced significantly by annealing temperature in an easy way. After 3 d of annealing, most PSN grains were $cube_{RD}$ oriented (Fig.4(c)), which is not typical PSN orientation. On the basis of the observations that the PSN at grain boundaries was more active and the total suppression of PSN inside some deformed matrices (Fig.2, position *A*), it is reasonable to consider that the $cube_{RD}$ orientation around particles should stem from grain boundaries. Besides, in contrast to the annealing at high temperatures leading to an increase of cube orientation even in grains at particles with annealing times[7], this was not the case at low temperature.

Although the number of PSN grains was larger than that of the grains formed at other sites, the effect of the latter on texture formation can not be ignored due to their size advantage. Fig.5 demonstrates the orientations of grains at grain boundaries (Fig. 5(a)) and the orientations of large grains (Fig. 5(b), regardless of whether they were in contact with large particles or not) after an anneal of 3 d (the orientations of PSN grains at this time are shown in Fig.4(c)). By comparing them, one can see that, firstly,



(a) (b) (c)

Fig.4 Evolution of microtexture of PSN grains of 82% rolled samples annealed at 330 ℃ for different times
(a)—40 s; (b)—16 h; (c)—72 h



(a) (b)

Fig.5 Orientations of grains formed at different sites (82%, 330 ℃, 3 d)
(a)—Grains at other sites; (b)—Large grains

the orientational differences between PSN grains and grains formed at other places are not large, indicating that many of them originated from the same type of nucleation site, possibly at grain boundaries; secondly, the orientations of large grains correspond to the $cube_{RD}$, $cube_{TD}$ and G components in macrotexture (Fig. 1(b)); thirdly, the nucleation at cube bands at low temperatures was ignorable in comparison with PSN and the nucleation at grain boundaries. The number of cube orientation was also carefully examined in the 97% rolled, 325 ℃ annealed sample which contained more cube grains when annealed at high temperatures[7], it was found to be very low (8%) too. Conclusively, both nucleation and growth of cube grains were not detected apparently in this temperature range (300 ~ 350 ℃).

4 DISCUSSION

It is frequently observed that PSN grains are small in comparison with the grains formed at other sites[4~6] because the growth of a PSN grain out of deformation zones is rather difficult due to the large opposing pressure (p_c) arising from its small radius of curvature[5] with respect to that of a grain formed at a planar grain boundary. If precipitation starts, the Zener pinning p_Z will add to the p_c to make the resisting force larger. The inhomogeneous precipitation will lead to an even larger difference in the grain sizes between PSN grains and those formed at other sites. According to the results obtained in this work as well as in Ref. [7], preferred PSN in C/S matrices was restricted by the favored precipitation in the same matrices leading to the small PSN grains. In contrast to this, the grains formed at grain boundaries could at least grow into the regions with few precipitates. Furthermore, the precipitation at grain boundaries gave rise to a low nucleation rate. Hence, the new grains had enough space to consume and were geometrically less inhibited by other grains. So, they became larger. This difference in grain size was solely caused by precipitation. Note that due to the strong precipitation and the geometrical obstacle of large particles which did not stimulate nucleation, the average grain size (13.7 μm) at 330 ℃ was not significantly larger than that at 450 ℃ (11.4 μm) as was the case in single phase alloy (25.9 μm at 350 ℃ and 15.3 μm at 450 ℃)[9]. Unlike those reported in previous works[1~6], at 300 ~ 350 ℃ the new grains could normally not overcome the pinning of fine precipitates, which was manifested by the absence of precipitates within new grains.

With respect to the influence of precipitation on recrystallization texture, a prevailing view point is that the precipitation will enhance the cube texture and reduce the texture caused by PSN, because the cube subgrains are larger than the subgrains with other orientations[4,6] and some cube subgrains are larger than the critical size for nucleation and therefore, growing directly before precipitation begins. This was only observed in the supersaturated single phase sample at high strain[11] and not in this two phase sample. Local orientation measurement by EBSD detected apparent cube orientation neither at the early stages nor at the later stages of annealing, not even in the 97% rolled samples. Although few cube grains and a coarse initial grain size in the initial sample could account for this, the real difference lies in that PSN proceeded, not more slowly, but faster at the early stages of annealing than cube grains, although their growth was significantly slower. As analyzed in Ref. [12], the $cube_{RD}$ and $cube_{TD}$ components are related with cube bands and can be the orientations of subgrains located at the border of cube bands. Thus, it is assumed that due to precipitation only the subgrains at the border of cube bands has enough time to grow before precipitation started, whereas the growth of cube subgrains in the center of cube bands is restrained by precipitation.

5 CONCLUSIONS

(1) Even under strong precipitation, there always exist two types of nucleation sites, namely, large particles and grain boundaries.

(2) An inhomogeneous precipitation and a bimodal particle distribution led to a size difference of recrystallized grains.

(3) The growth of PSN grains was even more strongly restricted than grains formed at grain boundaries due to the opposing pressure from its small radius of curvature and the Zener pinning. In contrast, the grains formed at grain boundaries had a size advantage and therefore contributed to the macrotexture.

(4) No preferred nucleation of cube grains due to precipitation was observed. Furthermore, as anneal went on, no apparent cube orientation was detected around particles as was the case of annealing at high temperatures.

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