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The Effect of Concurrent Precipitation on the Recrystallisation Texture of an AlMn-alloy

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Abstract

The present work reports on the effect of precipitation on recrystallisation of a cold rolled supersaturated Al1wt%Mn-alloy. It is observed that the precipitates form on the grain boundaries in the early stages of annealing under given conditions. It is further observed that these precipitates reduce the total number of successfully nucleated grains quite dramatically during recrystallisation. Particle stimulated nucleation of recrystallisation (PSN) is found to play an important role in the nucleation of grains comprising P- and ND-rotated cube orientations, which are the dominating texture components in case of concurrent precipitation. A nucleation mechanism for these texture components based on all processing steps is suggested.

1. Introduction

AlMn-alloys are among the oldest Al-alloys, but even today the alloy system is not completely understood. Due to its very good combination of strength, corrosion resistance, formability and brazeability, AlMn-alloys have been used in the heat exchanger industry for decades. For alloys in a supersaturated condition, which often is the case for AlMn-alloys, precipitation is known to retard nucleation and growth of the recrystallised grains, if the precipitates form prior to or during recrystallisation [1]. The grain structure shifts from being equiaxed towards being elongated in the rolling direction in the case of concurrent precipitation. Previous work has demonstrated that the P- and ND-rotated cube textures become the dominant orientations in the case of concurrent precipitation in an AlMn-alloy (AA3103) cold rolled to large reductions, typically $\varepsilon > 1.5$ [2,3]. The amount of necessary deformation depends on the initial texture. If a strong cube texture is present prior to cold rolling, *e.g.* as in hot rolled materials, then a higher deformation is required than for a randomly oriented material. Previous TEM-work [4,5] strongly indicate that these texture components are nucleated by particle stimulated nucleation (PSN).

By different post-processing procedures there are several possible ways to achieve different supersaturations of elements in solid solution for non heat-treatable alloys. However, all methods require an annealing treatment to achieve different concentrations in solid solution. The most common ways to achieve different amounts in solid solution are either by different: (i) homogenisation treatments, (ii) hot rolling temperatures and/or rolling reductions, or finally by (iii) cold deformation and subsequent annealing. In the present work the first method is chosen, and by this treatment formation of dispersoids is more or less avoided. By varying the homogenisation treatments relatively large differences in solid solution are achieved, and thus the effect of different solid solution concentrations on the softening behaviour, recrystallisation texture and grain size can be investigated.

2. Experimental

A commercially cast AA3103-alloy, containing 1.0%Mn, 0.57%Fe, 0.12%Si and other <0.02%, was given three different homogenisation treatments to ensure material conditions with different concentrations of Mn in solid solution. These amounts were calculated from electrical conductivity measurements, and the amounts were 0.5, 0.42 and 0.31 wt% for the respective homogenisation treatments, denoted A, B and C, respectively. The homogenisation treatments were as follows:

- *Homogenisation A*, heated and held at 610°C for 14h, then water quenched.
- *Homogenisation B*, heated and held at 610°C for 14h, then slowly cooled to 550°C and held at this temperature for 16h and finally water quenched.
- *Homogenisation C*, as *Homogenisation B* + slowly cooled to 500°C and held for 24h, and finally slowly cooled to 450°C and held for 32h and water quenched.

The *as-cast* material*,* containing 0.8 wt% Mn in solid solution has also been included in this work. The *as-cast* and homogenised materials were then cold rolled to true strains of 0.5, 1.5 and 3.0, followed by isothermal annealing at different temperatures. The softening and precipitation behaviour were followed by means of hardness and conductivity measurements. From these measurements it is possible to construct so-called time-temperature-transition (TTT) diagrams for the different material conditions [6]. The TTT-diagram of the A-material deformed to a strain of 3.0 is shown in Figure 1a), as this condition is the one analysed in detail in the present work. The full line indicates start of precipitation, while the broken lines indicate start and end of recrystallisation, respectively. A characteristic temperature, T_c, is indicated, at this temperature the precipitation curve crosses the curve indicating start of recrystallisation. Above this temperature (T_c), recrystallisation is being less affected by concurrent precipitation, while at temperatures below T_c heavy precipitation retards recrystallisation effectively. The characteristic temperatures of the different conditions are plotted in Figure 1b). From this figure it is seen that as the amount of manganese in supersaturated solid solution increases the characteristic temperature does the same. It is further seen that an increasing strain also raises the characteristic temperature for a given supersaturation of Mn in solid solution. The precipitation reaction was followed in detail for the A-homogenised material annealed below T_c with the use of a FESEM equipped with a solid state detector.

EBSD-mapping was carried out to identify the nucleation positions for recrystallised grains. In particular a close focus on the nucleation sites for the P- and ND-rotated cube orientations, which were found to be the dominating texture components in case of concurrent precipitation, was carried out.

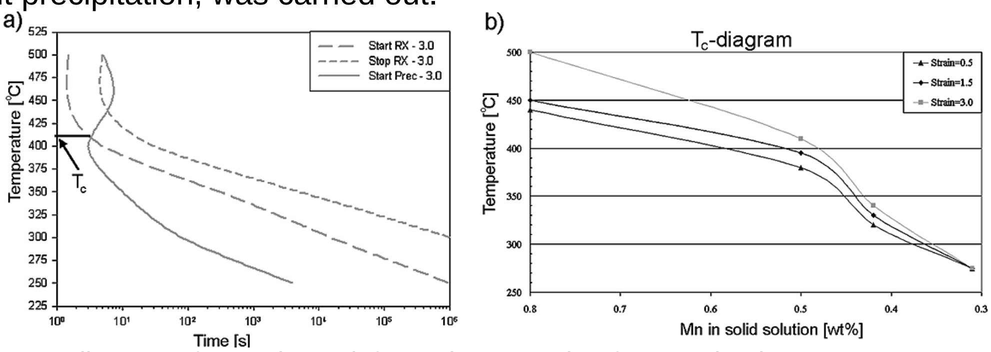

Figure 1: a) TTT-diagram of material A deformed to a strain of 3.0 and subsequently annealed at different temperatures. b) T_c-diagram of the different material conditions.

3. Results

With respect to the recrystallisation texture and grain size, the effect of annealing temperature is of greatest importance, as seen in Figure 2. When the annealing was carried out above the characteristic temperature, T_c, the fully recrystallised material consisted of small and equiaxed grains. However, for isothermal annealing below T_c, *i.e.* when precipitation retards both recovery and recrystallisation, the grains become larger and are elongated in the rolling direction. The recrystallisation texture also depends on whether the annealing was carried out below or above T_c. Below the characteristic temperature the recrystallisation texture was dominated by a strong P-texture in combination with a medium strength ND-rotated cube after deformation to a strain of 3.0. However, after a strain of 1.5 the intensity was not as strong. Annealing at temperatures above T_c generally resulted in relatively weak cube textures or random texture. The final textures represented by the φ_2=0°-section of the ODFs for the other investigated conditions are given in Ref. [3].

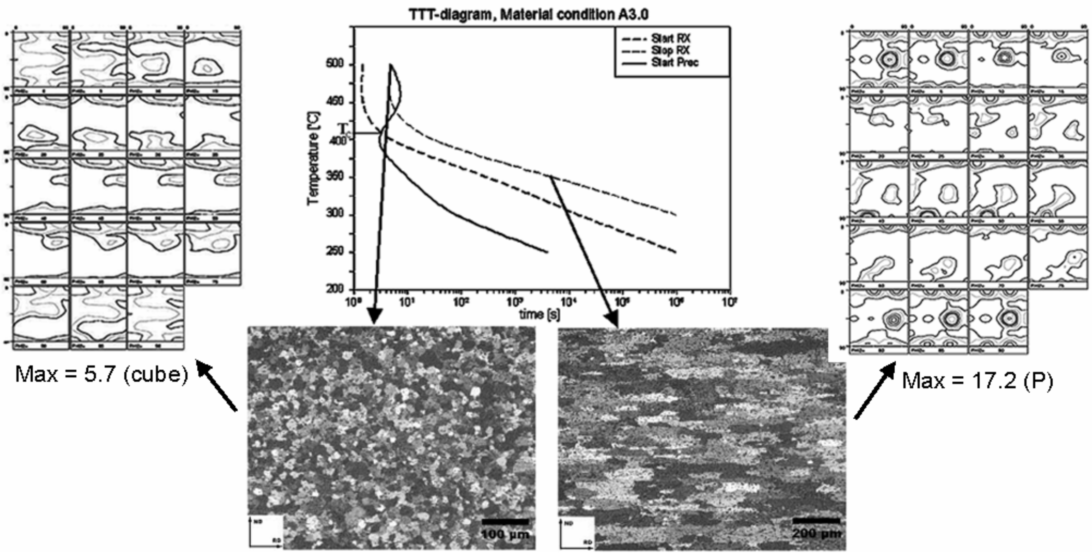

Figure 2: Effect of annealing temperature on the recrystallised grain structure and recrystallisation texture.

Figure 3a) shows the T_c-diagram and curves that indicate at what temperature the recrystallisation texture changes from random/weak cube texture to a combination of the P- and ND-rotated cube textures for a given supersaturation and deformation. It is seen that the curves follow the same trends. There are some minor differences, which could be due to too large temperature steps with respect to the annealing treatment or due to reading error of the graphs (hardness and conductivity). The overall conclusion is that the T_c-diagram, as given in Figure 1b), predicts the change in recrystallisation texture of a randomly textured material with a relatively high supersaturation reasonably well.

According to literature the P- and ND-rotated cube orientations are nucleated by PSN [2,4,5]. EBSD-mapping has shown that PSN certainly play a key role in the nucleation of these orientations for the present alloy as well, see Figure 3b). In this figure it is seen that the recrystallised grains with the actual orientations are positioned close to coarse primary particles (unindexed areas), which indicates that nucleation has occurred by PSN.

In Figure 4 micrographs of the A-material deformed to a strain of 3.0 and subsequently annealed at 350°C are given. The “large” white areas are particles (of which some are arrowed). Close investigation of these micrographs show that there are no dispersoids in the as-deformed condition, Figure 4a). After 60s of annealing the precipitates have formed (small white dots) and are positioned on the grain boundaries, as seen in Figure 4b). In Figure 4c), after 10 minutes of annealing, recrystallisation has started and one grain (texted bright area) is seen to have nucleated by PSN from the particle arrowed. A counting procedure of the dispersoids was carried out at the different stages of annealing and it was observed that the density was close to constant after an incubation time of approximately 60s. Even throughout the period where recrystallisation took place the density was more or less constant. This observation indicates that little or no precipitation occurred on the migrating high-angle grain boundaries (HAGB).

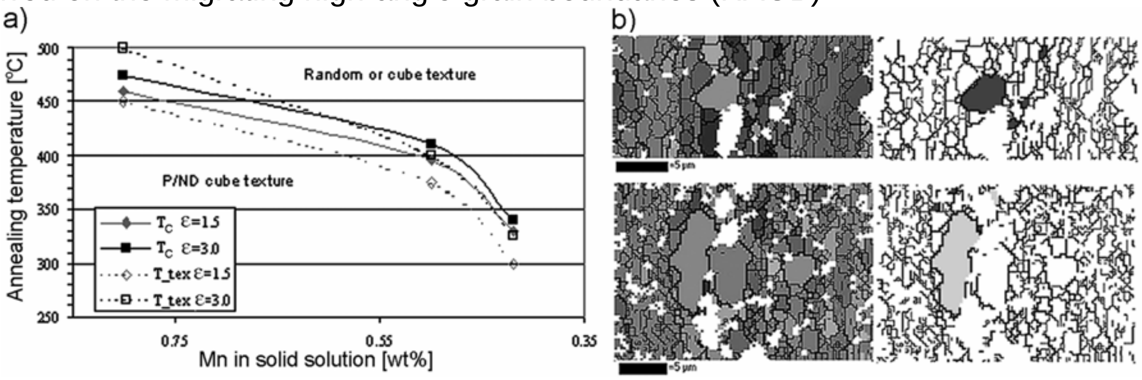

Figure 3: a) T_c-diagram and indication of at which temperature a change in recrystallisation texture is observed. b) OIM-maps of an annealed A-condition. Left figures show the raw map, including all grain boundaries, white areas are unindexed regions, most probably particles. Right figures show the grain boundaries and some shaded recrystallised grains comprising the P and ND-rotated cube orientations.

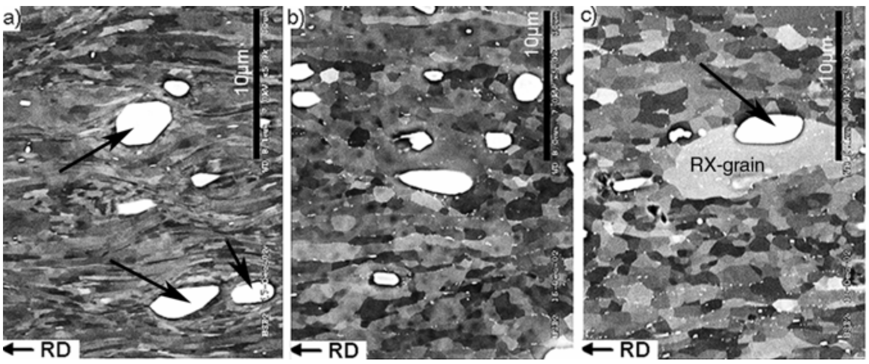

Figure 4: Microstructural evolution of the A-material a) deformed b) annealed 60s at 350°C and c) 10 min

4. Discussion

In the following, based on previous work and observations in the present work, the most likely origin of the strong P- and ND-rotated cube textures observed in the present work in case of concurrent precipitation is presented.

Homogenised material: According to theory depleted areas of elements in solid solution are formed close to the constituent particles during homogenisation. These areas become larger as homogenisation time increases. However, as the homogenisation time increases (A→B→C), the matrix becomes less supersaturated and consequently the total precipitation potential decreases.

Deformed material: Upon deformation large rotations of the original matrix are found close to the coarse particles. Previous work has shown that subgrains with P- and ND-rotated cube orientations, are present in the deformation zones if the deformation is large enough [9-11]. In the present work it was observed that if the other conditions were fulfilled, *i.e.* with respect to supersaturation and annealing temperature, a strain of ~1.5 was sufficient to produce a weak P-texture, *e.g.* Figure 3a). However, a strong and totally dominating P-texture was observed only for a strain of 3.0. This observation suggests that as the reduction increases, increasing fractions of subgrains obtaining these orientations are formed within the deformation zones. The amount of Cu-oriented deformation bands was also found to increase with increasing strain [12] and these bands have a 40°<111>-orientation relationship to the P- and ND-rotated cube orientations. This orientation relationship is known to give favourable growth conditions during recrystallisation.

Subgrain growth: During the first minutes of annealing a high density of precipitates formed. A lower dispersoid density was found within the deformation zones compared to the density in the matrix. This difference is associated with the lower supersaturation of Mn within the deformation zones. Subgrain growth is then expected to occur easier in the deformation zone due to the lower retarding forces acting against subgrain growth. Due to the presence of a strong recrystallisation texture it seems reasonable to assume that subgrains with P- and ND-rotated cube orientations, located within the deformation zones, grow on the expense of the deformed substructure, although not clearly why. At some point one or more subgrains consume the complete deformation zone, and are thus able to form nuclei for recrystallisation.

Recrystallisation: The decisive step to produce a recrystallised grain by PSN is the growth out of the deformation zone. It is believed that the 40°<111> rotation relationship to the Cu-orientation could result in a more effective nucleation for P- and ND-rotated cube orientations compared to those subgrains with no relationship between the subgrain and the matrix. With increasing deformation, the number of Cu-oriented deformation bands and the total number of subgrains within the deformation zones comprising P- or ND-rotated cube orientations increases. This results, as expected, in a more effective nucleation of these orientations as the deformation increases. Other nucleation sites than the PSN-sites are more effectively pinned due to the higher density of precipitates and higher amount of elements in solid solution that effectively slow down subgrain growth. The result is that other nucleation sites do not produce recrystallised grains as frequently as PSN. However, it should be noted that it is not only the P- and the ND-rotated cube orientations that are nucleated in case of concurrent precipitation. From detailed investigations on the growth of the recrystallised grains, it was observed that only about one out of five nucleated grains belong to either the P- or ND-rotated cube orientation [12]. These grains could be nucleated by PSN or by other nucleation mechanism like strain induced boundary migration (SIBM), but these grains mainly obtain a random orientation. Since the initial texture was close to random, and few cube-oriented subgrains were present in the deformed microstructure, nucleation from cube-bands that have survived deformation is probably not a significant nucleation mechanism in this study.

The elongated recrystallised grains found in case of concurrent precipitation are a result of a larger Zener drag in the normal direction (ND) than in the rolling direction (RD). Since there are more highly misoriented grain boundaries in ND and because of the fact that precipitation occurred on the boundaries prior to recrystallisation this leads to different Zener drags in the two directions. The difference in retarding forces results in an elongation of the grains in RD. A schematic illustration of this effect is given in Figure 5.

The reason why nucleation of the precipitates occurred on the grain boundaries is most probably due to the fact that as deformation increases, the solute atoms become attached to the boundaries and stay attached to these as the original grains become more and more elongated. This results in a higher concentration of solute atoms within the boundaries compared to the matrix. Hence, due to the difference in concentration, precipitation will occur easier on the boundaries than in the matrix during the subsequent annealing. For a binary Al1.3Mn-alloy Somerday and Humphreys [7] have shown that the precipitation mainly occurred on the original high angle grain boundaries (~85%). This observation has been verified for the present alloy as well, see Ref. [8]. These observations support the idea of more effective pinning of other nucleation sites like SIBM than the PSN-sites. For recrystallisation to occur a HAGB has to be present and since a high density of dispersoids is found at these, the nucleation event is effectively retarded at these positions. For PSN to occur, a HAGB is produced by the subgrain growth within the deformation zone. Hence, as a subgrain, growing from a region close to the particle interface, reaches the outer region of the deformation zone the misorientation to the matrix typically exceeds 30° for the reductions treated here, *i.e.* ε =1.5 and 3.0.

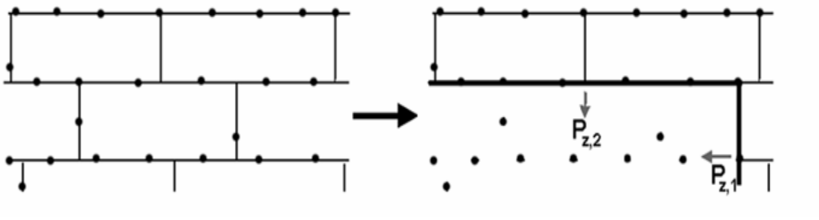

Figure 5: Schematic illustration of the effect of precipitation on recrystallisation. Thin lines are substructured regions, while thick lines represent migrating HAGB and dots are precipitates.

5. Conclusions

For a highly supersaturated AlMn-alloy cold rolled to large reductions precipitation of dispersoids occurs on grain boundaries, and mainly on those having a high-angle character (>15°). For certain conditions the precipitates form prior to recrystallisation, and thus act as a retarding force against recrystallisation, resulting in a slower softening kinetics in these cases. With respect to recrystallisation texture, the P- and ND-rotated cube orientations were found to be the dominating texture components in case of concurrent precipitation. A possible nucleation mechanism is suggested, however the mechanism is complex and all aspects are most probably not included.

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