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Abnormal grain growth in Al of different purity

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The transition from normal to abnormal grain growth has been studied in four Al alloys of various purity (2N, 3N, 4N and 5N). The temperature and time for the onset of abnormal grain growth depend strongly on the deformation and homogenization treatment. Generally, the formation of large grains before cold rolling makes easier the transition to abnormal grain growth during the subsequent annealing. The abnormal grain growth can take place only above a certain temperature which decreases with increasing alloy purity. The onset time of the abnormal grain growth decreases with increasing temperature. It can be qualitatively explained by the dissolution of submicron particles of a second phase. © 1998 Published by Elsevier Science Ltd. All rights reserved.

Keywords: grain growth; abnormal growth; grain boundary phase transitions

Introduction

The use of aluminium based alloys steadily increases. One of the important industrial problems is the *improvement of the surface quality* of several products which depends critically on the *uniformity of their grain structure*. The formation of very large grains or a large scatter of the grain size in a material can evoke a non-uniform deformation and recrystallization texture and, therefore, can cause intolerable shade fluctuations of the end product. The formation of a heterogeneous grain structure at any step of the aluminium transformation, from homogenization to intermediate annealing, makes the material unacceptable. Unfortunately, nowadays there exists no technology which would not allow a spontaneous formation of very large grains (so-called abnormal grain growth). This phenomenon has been well known since the nineteen-thirties[1–4]. However, the tendency to use pure aluminium for the production of industrial alloys, especially for deep drawing, has reviewed the old problem of control and/or suppressing the abnormal grain growth

Experimental

The materials used in our studies were 5N (99.999 wt.%), 4N (99.99 wt.%), 3N (99.92 wt.%) and 2N (99.00 wt.%) Al alloys. The main impurities in these materials are listed in *Table 1*. The alloys were produced by Pechiney CRV as hot rolled blocks 25 × 25 × 2 cm. The blocks are cut out, homogenized in air (Table 2), machined and cold rolled to a thickness of 2.5 mm with a reduction of 63–90% and a high number of passes (over 20). During the rolling the plates are periodically cooled in liquid nitrogen in order to keep their temperature below about −10°C and to prevent the recrystallization reaction. The cold rolled bands of 4N, 3N and 2N Al were annealed in order to obtain a fully recrystallized structure without a deformed matrix (with a mean grain size of 200–500 μm). The 5N material was annealed at 350°C for 30 min in order to prevent the grain growth. The regimes of homogenization and recrystallization annealings are listed in *Table 2*.

The recrystallized bands were cut into pieces with the dimensions 4 × 6 cm. These pieces were then annealed in an air furnace at various temperatures from 350 to 650°C. Some specimens were annealed only once and others were annealed several times at the same temperature in order to avoid the influence of repeated etching. After annealing the samples were etched for 1–2 min in a solution of 10 ml HF, 15 ml HCl and 90 ml H_2O in order to reveal the grain structure. The microstructure was photographed, and the mean grain size d was determined on 400–500 grains by the intersection method, using image analysing optical microscopy and polarization contrast. The measurements were repeated after each new annealing in the same area in order to diminish the influence of the difference of the starting grain size.

Results and discussion

The most important feature of the grain growth in the Al alloys studied is the *transition from normal to abnormal grain growth*. At the beginning of annealing, the

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Table 1 The main impurities in the Al alloys studied (concentrations are given in wt. ppm (10^{-4} wt.%))

Alloy	Fe	Cu	Si	Mg	Mn	Cr	Zn	Ti	Ga	Total
5N Al	2	1	7	1	< 1	1	< 4	< 1	2	10
4N Al	10	40	7	9						100
3N Al	170	40	300	10	10			240		770
2N Al	5000		1600		37					10 000

Table 2 The thermal treatment of the alloys studied

Material	Homogenization	Recrystallization
5N(1)	No	10 min, 350°C
5N(2)	70 h, 650°C	10 min, 350°C
4N(1)	No	10 min, 450°C
4N(2)	30 h, 570°C	10 min, 450°C
4N(3)	70 h, 650°C	10 min, 450°C
3N(1)	No	10 min, 450°C
3N(2)	30 h, 570°C	10 min, 450°C
2N	30 h, 570°C	10 min, 450°C

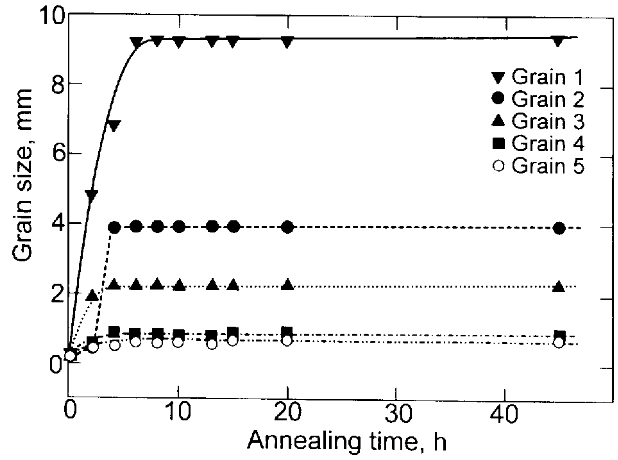

Figure 2 Time dependence of the mean grain size in 4N Al at 500°C (homogenization at 650°C for 70 h) measured in various 'old grains'

grain structure is uniform and the scatter of the grain size is low. Nevertheless, the cold rolled microstructure reflects the grain structure existing already before the deformation. The newly recrystallized grains are homogeneous inside an old grain of the former matrix, but the mean grain size varies greatly between the old grains. Later, the new recrystallized grains grow inside the old grains. For some of them, the growth is faster and some big new grains appear, having often the same shape as the former old grain (*Figure 1*). Thus, inhomogeneity develops, and the grain size distribution starts to be bimodal.

When changing the homogenization regimes (*Table 2*), we have observed that the size of the 'old grains' formed in the sample after the homogenization, prior to the cold rolling affects critically the onset of the abnormal growth. The larger the size of the 'old' grains, the easier the transition to the abnormal grain growth. Generally, the 'old' grain boundaries, i.e. the borders between the colonies of the new recrystallized grains, stop effectively the abnormal grain growth. The abnormal growth in different colonies ('old grains') starts after different time (*Figure 2*).

Even in the case of very large recrystallized grain colonies (big 'old grains'), there exists a temperature, below which a stagnation of the normal grain growth occurs, without transition to abnormal growth. This feature was already observed earlier[5,6]. The data plotted in *Figure 3* show that the temperature of the onset of abnormal grain growth decreases with increasing purity of the alloys studied. The data[7] for the 99.6 wt.% Al fit that curve well.

At a constant impurity content, the onset time of the abnormal growth decreases with increasing temperature (*Figure 4*) at least for 3N Al. This dependence can be explained by the dissolution of submicron intermetallic particles during the grain growth. During the homogenization at the temperature close to the melting point, all precipitates dissolve in the solid solution. New precipitates can build during the maintaining at room temperature and cold working (most possibly Al_3Fe because the Fe content in 3N Al is above the solubility of Fe at $T > 560°C$[8]). The grain boundaries fixed at the precipitates can become free during the grain growth, according to Zener's idea, due to the partial dissolution of ankering particles. According to[9], the radius r of the precipitate changes with the dissolu-

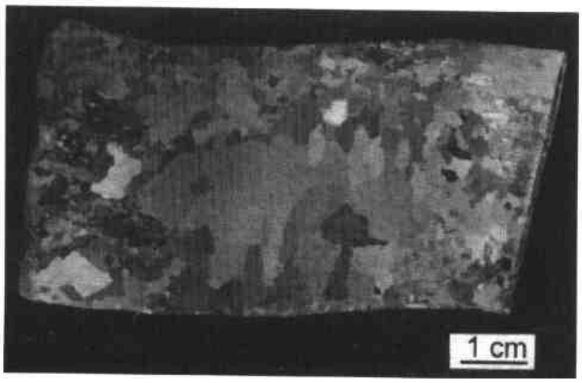

Figure 1 Microstructure of a sample after the onset of abnormal grain growth. 5N Al, homogenization at 650°C for 70 h; deformation 63%; thickness 2 mm; annealing at 450°C for 5 min

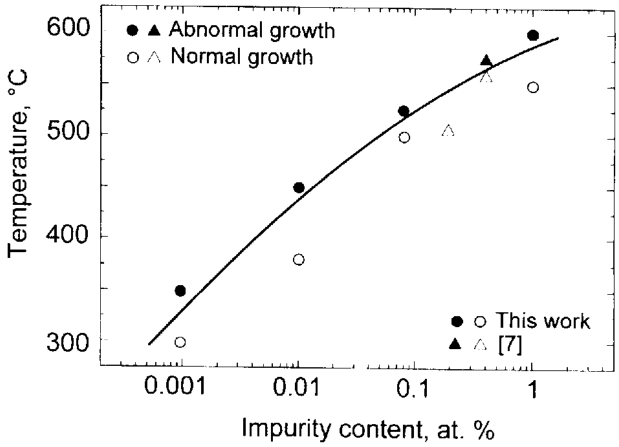

Figure 3 Dependence of the barrier temperature for the abnormal grain growth on the impurity content of the Al alloys studied. The literature data for the 99.6 wt.% Al are also presented[7]

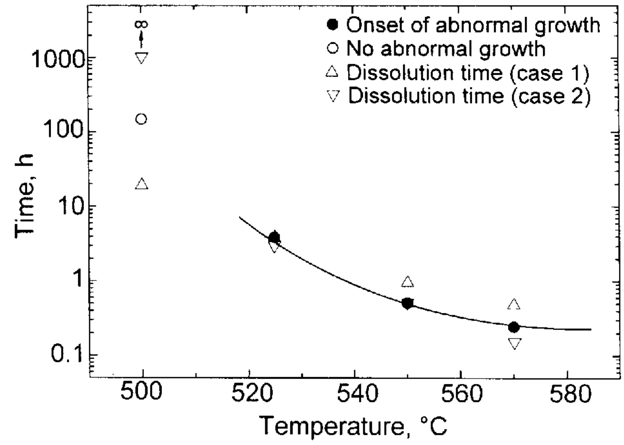

Figure 4 Temperature dependence of the onset time for abnormal grain growth in 3N Al and of the calculated dissolution times for the Al_3Fe precipitates

tion time t as follows:

$$r^2 = r_0^2 - 2\alpha D_m t,$$

where r_0 is the starting radius of a precipitate, D_m is the Fe diffusivity of Fe in Al[10] and

$$\alpha = (c_0 - c)/(c_{Al_3Fe} - c_0)$$

where c is the Fe concentration in the solid solution, c_0 is the Fe solubility at the annealing temperature, and c_{Al_3Fe} is the Fe content in Al_3Fe. One can estimate the time of the complete dissolution of a particle, having the radius r_0 before annealing: $t = r_0^2/(2\alpha D_m)$. In *Figure 4* the estimation data are shown for case 1 ($r_0 = 0.2$ μm, $c = 20$ wt. ppm) and case 2 ($r_0 = 0.1$ μm, $c = 57$ wt. ppm). If $t \to \infty$, no abnormal grain growth can happen. The estimations from this simple model fit well the experimental data on the onset time of abnormal grain growth (*Figure 4*).

Conclusions

The following conclusions can be drawn from the present studies.

- The time and the temperature of the onset of abnormal grain growth depend strongly on the deformation and homogenization treatment.
- In the alloys studied, the abnormal grain growth proceeds only above a certain temperature which decreases with increasing alloy purity.
- The onset time of the abnormal grain growth decreases with increasing temperature.
- This decrease can be qualitatively explained by the dissolution of submicron particles of a second phase.
- The abnormal grain growth in high-purity Al can be suppressed with the aid of a suitable combination of deformation, heat-treatment and micro-alloying.

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