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The Onset of Abnormal Grain Growth in Al-Ga Polycrystals

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Abstract. The grain growth has been studied in high-purity Al and Al–Ga alloys with Ga contents of 10 wt. ppm, 50 wt. ppm and 1 wt.%. The transition from normal to abnormal grain growth has been investigated in dependence on Ga content, temperature and sample thickness. Below ≈370°C only normal grain growth occurs. Above this temperature a very sharp transition to abnormal grain growth is present in all materials studied. The influence of the orientations of individual grains and the grain boundary mobilities on the onset of abnormal grain growth is discussed.

1. Introduction

The studies of individual grain boundaries (**GB**s) in bicrystals show that the GB mobility *m* can differ by many orders of magnitude, depending the GB misorientation and the impurity concentration [1]. Theoretical calculations and computer simulation of grain growth in polycrystals predict that if the mobility ratio between „slow" and „fast" GBs reaches a certain amplitude (about 5) the transition from normal to abnormal grain growth can occur [2,3]. The influence of GBs misorientations on the onset of abnormal grain growth can be analysed with aid of the modern methods of electron microscopy which permit to measure the misorientation parameters of individual GBs in polycrystals. The goal of this work was to investigate the transition from normal to abnormal grain growth in pure Al and Al–Ga alloys in dependence on Ga content, temperature and sample thickness.

2. Experimental

The 5N (99.999 wt.%) Al and three Al–Ga alloys containing 0.001, 0.005 and 1 wt.% Ga were investigated. The alloys were produced from 5N Al and 5N Ga as ingots with a weight of about 7 kg. The foundry state products are cut out, homogenized (30 h at 570°C), machined and cold rolled to a thickness of 2.5 and 0.6 mm with a reduction of 60% and a high number of passes (over 20). During the rolling the plates are periodically cooled in liquid nitrogen in order to prevent recrystallization. The cold rolled bands were annealed at 450°C for 5 min in order to have a fully recrystallized structure without deformed matrix (with a mean grain size of 200–500 mm). The recrystallized bands were cut into pieces with the dimensions 4 on 5 cm. These pieces were then annealed in an air furnace at the temperatures of 350, 380, 405 and 425°C. Each specimen was annealed several times at the same temperature. After each break the sample was etched for 1–2 min in a solution of 10 ml HCl, 50 ml HNO_3 and 50 ml HCl in order to reveal the grain structure. The microstructure was photographed and the mean grain size *d* was determined on 400–500 grains with aid of an intersection method using the optical microscopy. The measurements were repeated after each new anneal in the same area in order to diminish the influence of the difference of the starting grain size.

3. Results

At the beginning of the grain growth anneals the samples have the „starting" microstructure after the recrystallization annealing at 450°C for 5 min. This microstructure varies according to the alloys. The microstructure of 5N Al and alloys containing 0.001 and 0.005 wt.% Ga was homogeneous and it is easy to determine the mean grain size of the new recrystallized grains, which generally is about 500 μm. On the contrary, the microstructure of the Al–1 wt.% Ga is very heterogeneous. The recrystallization occurs

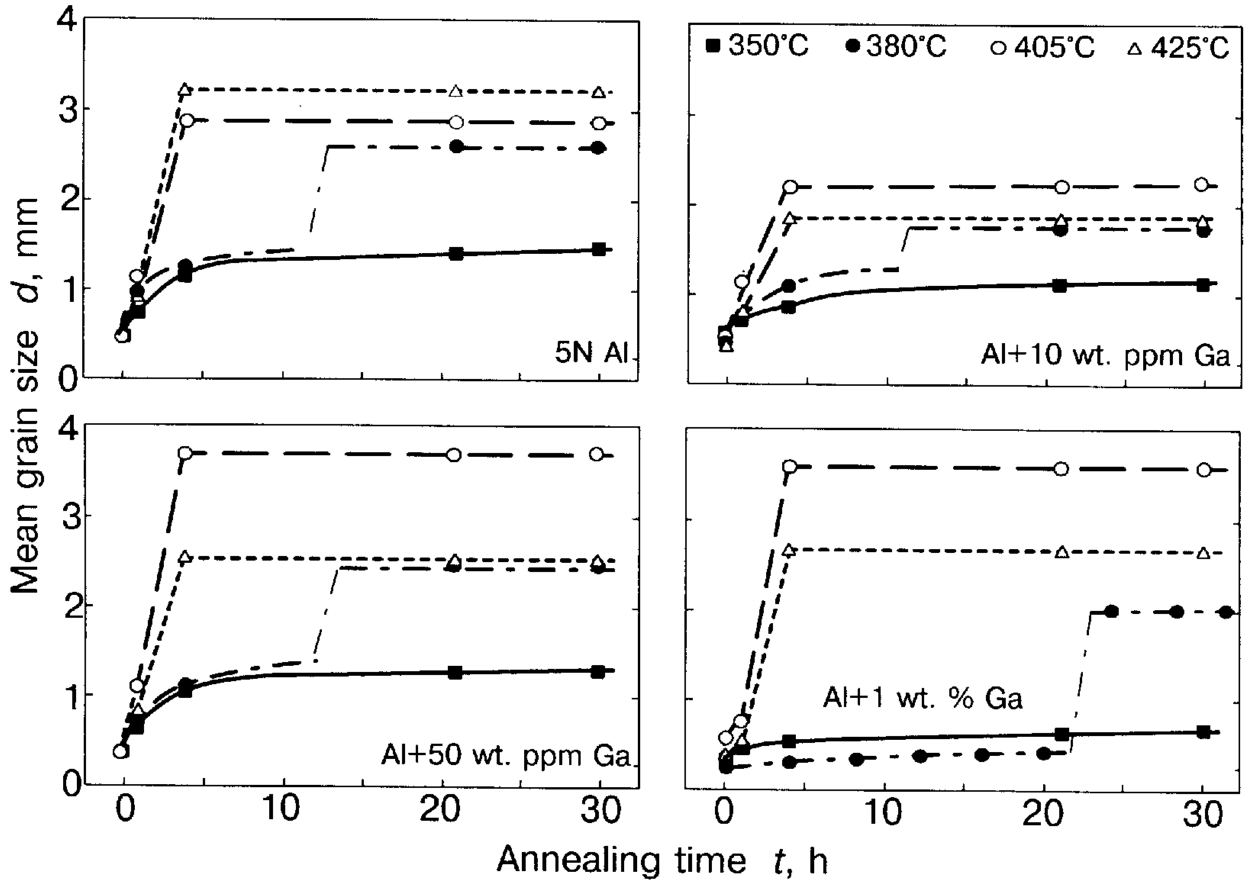

Fig. 1. The dependencies of the mean grain size *d* on the annealing time for 5N Al and three Al–Ga alloys at different temperatures. The sample thickness is 2.5 mm.

inside the old grains of the matrix and, therefore, the outline of the old grains can still be seen. Consequently, the recrystallized new grains in the Al–1 wt.% Ga are homogeneous in each old grain but strongly differ between the different old grains (grain size 200–800 μm). In Fig. 1 the dependencies of the mean grain size *d* on the annealing time *t* for 5N Al and three Al–Ga alloys at four different temperatures are shown. In Fig. 2 the influence of the sample thickness on *d(t)* dependencies is shown. At 350°C only normal grain growth occurs. The equiaxial grains grow uniformly. The grains growing abnormally fast do not appear even after 100 and 720 h (these points are not shown in the figure). The growth rate diminishes with *t*. The fastest grain growth occurs in the pure metal. The alloys containing 0.001 or 0.005 wt.% of Ga grow slower and reach a mean grain size which is slightly below that one of 5N Al. The mean grain size for Al–0.005 wt.% Ga is larger than the value for Al–0.001 wt.% Ga. The grain growth is clearly slowed down in Al–1 wt.%Ga. At higher temperatures we observe a sudden and strong grain growth after the stage of the normal grain growth. The mean grain size is now about 2–3 mm and some grains are larger than 5 mm. Between 10 and 20 h of annealing at 380°C an acceleration of the grain growth is observed. This phenomenon is weak in Al–0.001 wt.% Ga, more important in pure Al and Al–0.005 wt.% Ga, and very strong in Al–1 wt.% Ga. The shape of the curve is so modified that we have to call it abnormal growth. After 20 h of annealing, the grains do not grow any further for an annealing time of 70 h. This abnormal growth does not only occur in Al–Ga alloys but also in 5N Al, whose mean grain size is still larger in comparison with the Al–Ga alloys. The higher the temperature of annealing, the faster the grain growth and the larger the mean grain size. At 405 and 425°C this abnormal grain growth is so strong that already after several hours a stagnation is reached. Whereas during the first hour of annealing the mean grain size grows from 500 up to 800 μm, during the next three hours it grows from 800 mm up to 3 mm. The abnormal grain growth occurs during this period of annealing and the phenomenon is stronger and earlier than at 380°C. The same behaviour is observed in the pure metal and in the alloys containing 0.001 or 0.005 wt.% Ga.

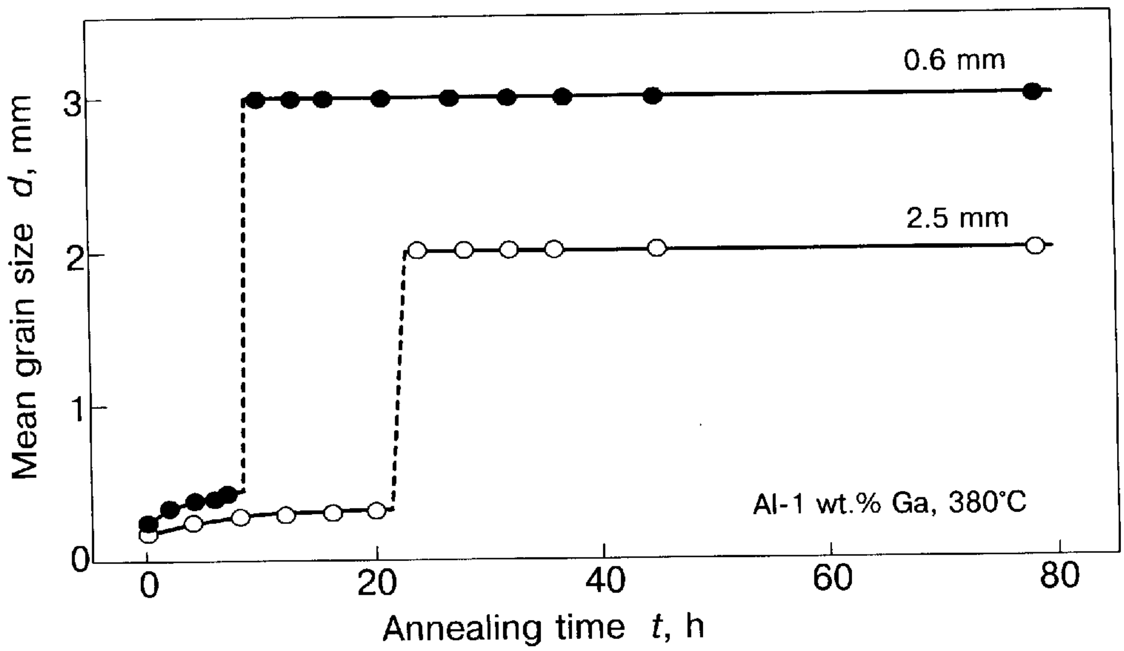

Fig. 2. The dependencies of the mean grain size d on the annealing time for the samples of different thickness.

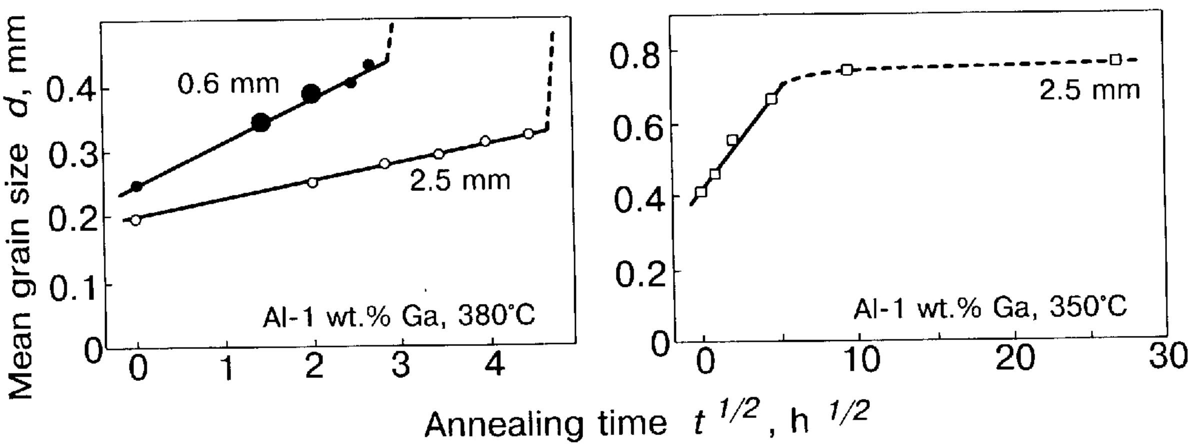

Fig. 3. The dependencies of the mean grain size d on the square root of annealing time at the stage of normal grain growth.

4. Discussion

During normal grain growth the size of individual grains is relatively uniform and the grain size distribution (i.e. the grain size d normalized by the mean grain size d_0) does not vary with time t. Normally, d_o -t^n where n should be equal to 0.5 [4, 5]. In some conditions normal grain growth changes abruptly to the so-called secondary recrystallization or abnormal grain growth [6, 7]. In this case few grains suddenly begin to grow very fast at the expense of all other recrystallyzed grains until these are consumed. In our experiments the addition of Ga slows down the grain growth at the stage of normal grain growth. The fastest grain growth occurs in the pure metal. In the alloys containing 0.001 or 0.005 wt.% Ga the grains grow slower and reach a mean grain size which is slightly below that one of 5N Al. The grain growth is clearly slowed down in Al–1 wt.% Ga. These results are in line with the impurity drag theory [6, 8, 9] This theory predicts that the impurity atoms drag the grain boundary migration and, therefore, decelerate grain growth.

Above a certain temperature the transition from normal to abnormal grain growth occurs in pure Al and in the Al–Ga alloys studied. This transition is very sharp if the mean grain size is plotted as a function of time. For example (see Fig. 2), the onset time of the abnormal growth for a sample with thickness 0.6 mm lies between 7 and 10 h. After 7 h the fine grained matrix is exclusively present. Three hours later virtually only large abnormal grains remain in the sample. The temperature influence on the onset of the abnormal grain growth is also very pronounced:

- At 350°C abnormal grain growth do not proceed even after 720 hours;
- At 380°C abnormal grain growth begins after 20 hours;
- At 425°C abnormal grain growth begins after about 2 hours and the stage of normal grain growth practically disappears.

Therefore, a relative narrow temperature interval exists (between 350 and 425°C) where the transition from normal to abnormal grain growth can be observed. Below this interval the grains grow slowly and the grain structure remains fine. Above this narrow temperature interval the large grains appear whithin a very short time and it is practically impossible to conserve a fine grain structure.

Secondary recrystallization plays a very important role in defining the microstructure and texture of many technologically important materials [8]. In these materials the secondary recrystallization begins after the suppressing of the normal grain growth by pores or fine particles of a second phase. Such an inhibition of the normal grain growth was assumed to be a necessary condition for the secondary recrystallization [7]. In our experiments, normal grain growth is inhibited only at 350°C (see Figs 1 and 3). If abnormal growth proceeds, d is propotional to the square root of the annealing time until the onset of the abnormal growth (see Fig. 3). Therefore, in our case the inhibition of normal growth is not necessary for the beginning of abnormal growth. The present theory [2] predicts different conditions for the onset of abnormal growth in three-dimensional and the two-dimensional polycrystals. The conditions for the grain boundary mobility and energy ratios between normal and abnormal grains, necessary for the beginning of the abnormal growth, are less rigid for a two-dimensional grain structure. It could be supposed that abnormal growth is triggered in the areas where the grains reach both sample surfaces and the grain structure becomes two-dimensional. Really, in our experiments abnormal growth begins earlier in the thinner polycrystal (see Fig. 2). But at the beginning of abnormal growth, d is smaller than the sample thickness. We have measured the misorientation parameters of GBs in the normal matrix and among the grains contacting with the first large grains at the very beginning of abnormal growth [10]. The orientation of the normal grains is close to (112). Many of abnormal grains have different surface orientation close to (110) or (100). If the number of grains contacting with free surface is large enough, the difference in surface energies among neighboring grains can also trigger abnormal grain growth. In this case abnormal grain growth should begin earlier in the thinner polycrystal even if the grain structure is still three-dimensional.

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References

[1] C.H.Wörner, P.M.Hazzledine, Mat. Sci. Forum **113–115**, 379 (1993).
[2] G.Gottstein, L.Shvindlerman, Acta metall. mater. **41**, 3267 (1993).
[3] A.D.Rollett, D.J.Srolowitz, M.P.Anderson, Acta metall. **37**, 1227 (1989).
[4] I.M.Lifshitz, J. Exp. Theor. Phys. **42**, 1354 (1962).
[5] V.E.Fradkov, L.S.Shvindlerman, Structure and Properties of Interfaces in Metals (Nauka Publishers, Moscow, 1988) p. 213 (in Russian).
[6] G.Gottstein, Rekristallisation metallischer Werkstoffe (DGM, Oberursel, 1984).
[7] V.Ju.Novikov, Secondary Recrystallization (Metallurgia Publishers, Moscow, 1990) (in Russian).
[8] S.S.Gorelik, Recrystallization of Metals and Alloys (Metallurgia Publishers, Moscow, 1978) (in Russian).
[9] F.Haessner, Recrystallization of Metallic Materials (Dr.Riederer Verlag, Stuttgart, 1978).
[10] V.Sursaeva, B.Straumal, S.Risser, B.Chenal, W.Gust, L.Shvindlerman, phys. stat. sol. (a) **149**, 379 (1995).