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Normal and Abnormal Grain Growth in Tungsten Polycrystals

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ABSTRACT

The grain growth has been studied in high-purity flat W polycrystals at 2000°C in vacuum. The onset of abnormal grain growth proceeds without inhibition of the normal grain growth. After appearing of the large abnormal grains the areas with small "normal" grains exist very long. The polycrystal has a bimodal grain structure, both normal and abnormal grains continue to grow. The orientations of individual grains were determined with aid of the electron backscattering diffraction method in the contact area between the abnormally growing large grains and normal matrix. In samples of a larger thickness the abnormal grain growth do not appear in the time period studied. The influence of the formation of new grain boundaries with a high mobility on the onset of abnormal grain growth and similarities with the abnormal grain growth in Al alloys are discussed.

INTRODUCTION

The formation of very large grains or a large scatter of the grain size in a material due to the onset of the abnormal grain growth can deteriorate drastically the properties of the material. The studies of individual grain boundaries (GBs) 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]. Recently the transition from normal to abnormal grain growth in pure Al and Al–Ga alloys in dependence on Ga content, integral concentration of impurities, temperature and sample thickness was studied [4–6]. It was shown that a decreasing thickness of the sample diminish drastically the onset time of the abnormal grain growth. Below a certain temperature the abnormal grain growth do not appear [6]. This barrier temperature decreases with increasing purity of Al [6]. It is important to prove whether these regularities established for Al, having a low-melting temperature and face-centred cubic lattice, are general enough. Therefore, the goal of this work was to investigate the grain growth in a metal with a high melting point and a body-centred cubic lattice. As an object for this investigation W polycrystals were chosen. In this material the drastic influence of the GB misorientation on the GB wetting behaviour was recently demonstrated [7].

EXPERIMENTAL

Tablets of pure W compact (diameter 10 mm, height 5 mm) produced by Metallwerk Plansee GmbH were used. They were cutted with the aid of spark erosion in order to produce the specimens of different thickness *h* (0.8 mm and 2 mm). The samples were than annealed at 2000°C in vacuum

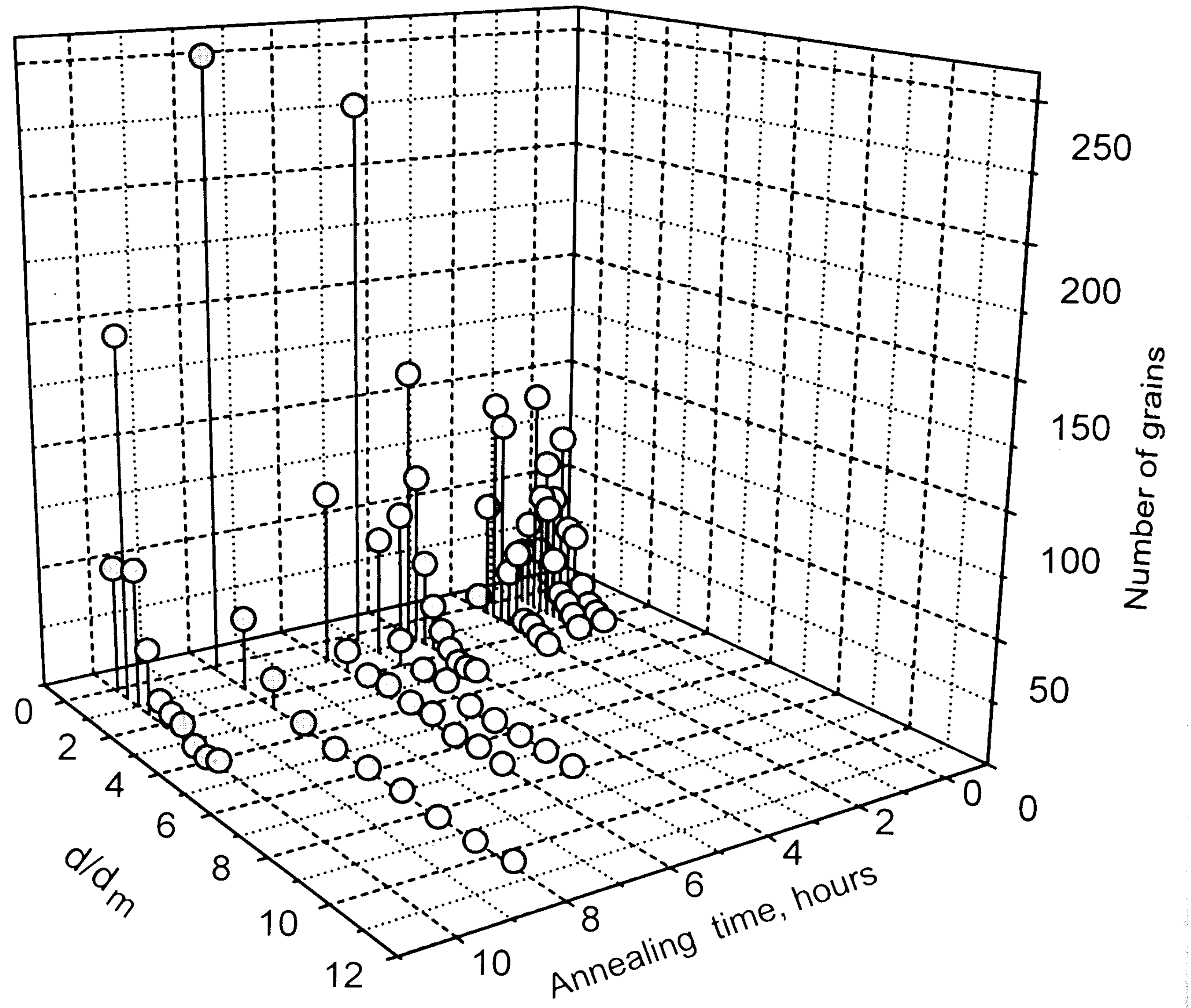

Fig. 1. Size distributions for various annealing times at $T = 2000°C$ for $h = 0.8$ mm, d is the grain size and d_m is the mean grain size.

of 10^{-8} Pa during various times from 0.5 to 10 h. For each anneal a separate sample was used. Already after 0.5 h the W polycrystals revealed the fully recrystallized structure without deformed matrix. After annealings the microstructure of the W polycrystals was investigated with the aid of scanning electron microscopy, optical microscopy and the electron back scattering diffraction technique. 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.

RESULTS AND DISCUSSION

Figure 1 displays the distributions of the grain size d for various annealing times t at $T = 2000°C$ for $h = 0.8$ mm. For each t the grain size is normalized to the mean grain size d_m. Figure 2 shows the time dependence of d_m in W polycrystals having different thickness h. At low t the normal grain growth proceeds. After $t = 5$ h very large grains appear in the polycrystal. The size distribution becomes bimodal. The transition from normal to abnormal grain growth happens. The onset of abnormal grain growth proceeds without inhibition of the normal grain growth. After appearing of the large abnormal grains the areas with small "normal" grains exist very long. The increase of

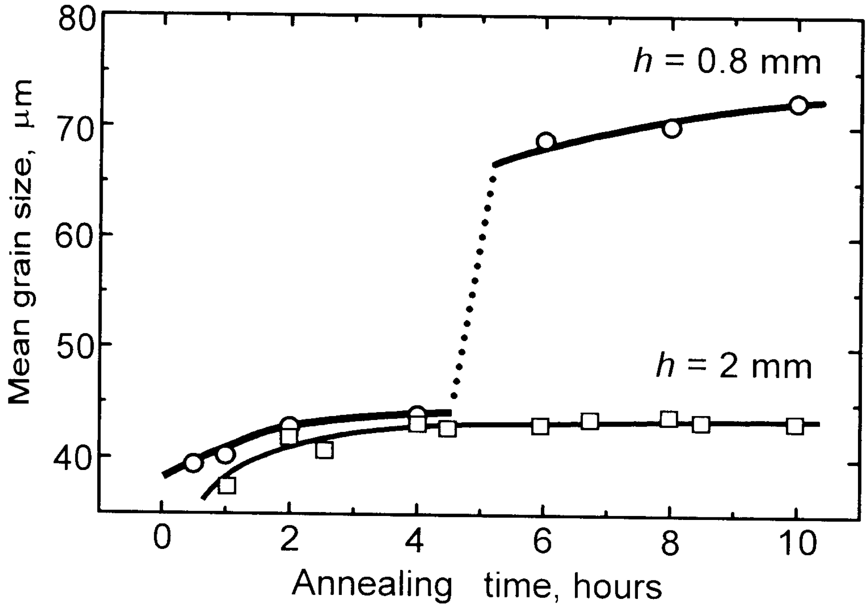

Fig. 2
Time dependence of the mean grain size in W polycrystals having different thickness *h*.

the mean grain size is accompanied by an increase of the grain size variation. It is also the sign of the abnormal grain growth. In the thicker sample the normal grain growth remains not interrupted, at least in the time interval studied. The transition to the abnormal grain growth do not proceed.

Figure 3 shows the inverse pole figure for a W polycrystal annealed at 2000°C for 10 h having a thickness h = 0.8 mm. The sample has an orientation texture. The axes <110> and <112> of about 50% of grains are nearly parallel to the normal direction (ND) to the sample surface. The analysis of microstructure demonstrates that the grains of <110> ND and <112> ND orientation are not randomly distributed but are clustered. Each cluster contains 5 or 6 grains separated by high-angle GBs.

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 [9]. 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 3-dimensional and the 2-dimensional polycrystals. Computer modelling of the grain growth revealed that the transition from the normal to the abnormal grain growth can start if the scatter of GB mobilities in the polycrystal is high enough, and the mobility of certain GBs is somewhat higher than the mean GB mobility [2, 3]. It was also shown that this barrier difference of GB mobilities is less in case of the 2-dimensional grain growth (all grains intersect both surfaces of the platelete) than in case of the 3-dimensional growth (only a part of grains contacts the free surface) [2, 3]. Recently it was observed in Al – 1 wt. % Ga alloy that the onset time of abnormal grain growth increases with increasing thickness of the sample [5]. The size of the grains growing abnormally fast was larger than the sample thickness and the grain size of the "normal" fine-grained matrix was smaller than the sample thickness. Therefore, it could be supposed that the transition from the normal to the abnormal grain growth is triggered by the transition from 3-dimensional growth to the 2-dimensional one. The indication for that was also that the fine-grained matrix was very fast consumed by the "abnormal" grains. In this work the increase of the sample thickness suppresses

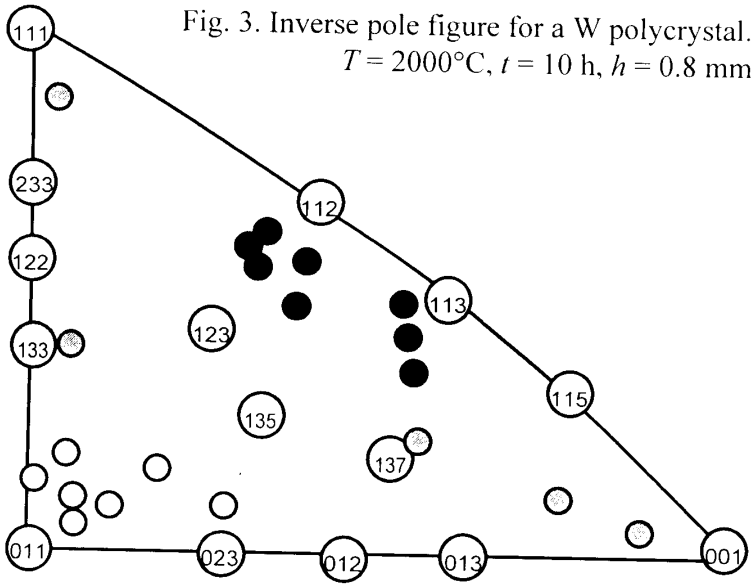

Fig. 3. Inverse pole figure for a W polycrystal. $T = 2000°C$, $t = 10$ h, $h = 0.8$ mm.

also the onset of the abnormal growth. But the abnormally large grains are nevertheless much smaller than the sample thickness. In other words, even after the onset of the abnormal growth the conditions for 3-dimensional growth are still valid. Therefore, the transition from normal to the abnormal grain growth in W is not triggered by the transition from 3-dimensional growth to the 2-dimensional one. The continuation of the normal grain growth in the fine-grained matrix after the onset of the abnormal grain growth confirms this conclusion.

The W polycrystals studied possess a clustered structure containing both small-angle GBs with low mobilities and high-angle GBs with high mobilities. Threfore, we can suppose that the barrier difference in mobilities needed to trigger the abnormal growth can be reached in case of W as a result of the GB misorientaion "summation" which proceeds during the gradual dissappearance of the smaller grains in the fine-grained matrix. Another reason for the transition to the abnormal growth can be the difference of the surface energies of grains having different orientations. In the W polycrystals studied the texture is rather pronounced. If the number of grains contacting with the free surface is large enough, the difference in the surface energies among neighboring grains can also trigger abnormal grain growth. In this case abnormal grain growth should begin earlier in thinner polycrystals even if the grain structure is still 3-dimensional.

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