

Effect of the Degree of Order and the Deformation Microstructure on the Kinetics of Recrystallisation in a Fe₃Al Ordered Alloy

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Abstract. A Fe₃Al based single phase alloy was rolled at 600 °C and 400 °C and the kinetics of recrystallisation in A2 and B2 phase regions was determined in conjunction with the kinetics of the A2-B2 ordering and recovery. At 700°C and 800°C the recrystallisation kinetics depends strongly on the temperature of rolling while at 900°C only a small influence is visible. These results are attributed to the high stored energy in the sample rolled at 400 °C and its high recovery rate at 900 °C, leading to a similar level in stored energy as for the sample rolled at 600 °C in the beginning of annealing. At lower temperatures, in contrast, the difference in stored energy is conserved over longer times in the annealing process. Almost half the apparent activation energy was observed in the ordered state compared to that in the disordered state.

Introduction

Fe₃Al based alloys with A2, B2 or D0₃ structures are candidates for high temperature structural materials in, for example, power plants and petrol-chemical plants because of their good corrosion resistance, low cost and relatively low density. Although many intermetallic compounds often show serious room temperature brittleness, Fe₃Al alloys show relatively good room temperature ductility in rolled and well-recovered states [1]. However, the ductility drops drastically by the occurrence of recrystallisation [1], which would lead to severe problems for applications. It is therefore of importance to understand and control the recrystallisation behaviour.

Since the critical temperatures for the A2-B2 disorder-order transition (second-order transformation) and the B2-D0₃ order-order transition is in the same temperature range as recrystallisation, some investigations have been done on the effect of the degree of order on recrystallisation and recovery [2, 3]. Morris et al. [2] especially studied it in the B2 and D0₃ ordered phase regions and observed no significant effect of the D0₃ ordering on the kinetics of recrystallisation. They explained that the reason could be the low degree of order (0.3-0.4) or the gradual increase of the order below the critical temperature due to the second-order transformation. Huang et al. [3] investigated recovery and recrystallisation in the B2 and A2 phase regions and mentioned that the long range order retards the kinetics of grain growth based on the slight decrease in JMAK exponent by ordering. It is of interest to investigate this effect more in detail.

It is also interesting to determine the effect of deformation temperature on recrystallisation and recovery since deformation temperature greatly changes the deformation microstructure and subsequent recovery and recrystallisation kinetics. In the present study, warm rolling experiments at 600 °C in B2 region and 400 °C in D0₃ region and annealing experiments in A2 and B2 phase regions have been performed for a Fe₃Al based single phase alloy. Based on the experiments, the recrystallisation kinetics was determined in conjunction with the kinetics of the A2-B2 ordering and recovery.

Experimental Procedure

A Fe-26Al-5Cr alloy was prepared, which shows the critical temperature of the A2-B2 transition at 813 °C and the D0₃-B2 transition at 552 °C determined by DTA measurements. This material was at first hot-rolled and heat-treated to get fine-grained microstructure and then cut into blocks of 50 x 100 x 5 mm (TD x RD x ND). The blocks were subsequently warm rolled at 600 °C and 400 °C to a reduction of 73 % and 65 %, respectively, hereafter referred to as ‘600R sample’ and ‘400R sample’, respectively. The rolling experiments were performed on a laboratory rolling mill with 10~15 % reduction per pass, as described in detail in a previous paper [4].

Annealing was performed for samples cut into a size of 15 x 9 mm (TD x RD) at 900 °C in A2 phase region and 800 °C and 700 °C in B2 phase region for 10 seconds up to 10 hours in a salt bath, followed by water quenching. Heating rates were tried to be the same for all samples.

Deformed/annealed microstructures and textures were determined by automatic crystal orientation mapping (ACOM) which is based on the automatic analysis of electron backscattered diffraction (EBSD) patterns in high-resolution SEM. Microstructures were determined on a plane parallel to the rolling plane in the centre layer (*s*=0) of the specimens. The maps were measured with a step size of 2~20 µm on areas of approximately 10 x 5 mm². Recrystallised area fractions were measured by taking into account grain size, aspect ratio and internal misorientation of the grains. ACOM samples were prepared by mechanical grinding, polishing and electropolishing.

The degree of long range order and the amount of strain in the deformed and the annealed samples were determined by X-ray diffraction using CoK_α radiation. X-ray detection was performed with an area detector which allowed to measure the line profile of several diffraction peaks at the same time. The degree of order was estimated by texture measurements of the superlattice (200) and fundamental (400) peaks, using the following equation (1).

$$S_{B2} = ((I_{200}/I_{400})_{\text{exp}}/(I_{200}/I_{400})_{\text{ref}})^{0.5} \quad (1)$$

where ref is a sample that was well ordered by a heat treatment at 850 °C for 15 min followed by an annealing at 500 °C for 1 day and at 400 °C for 1 week, followed by furnace cooling [5]. The amount of strain was determined by half-width measurements of the fundamental (400) peaks fitted to Gaussian functions.

Results

Deformed Microstructures and Textures. Figure 1 shows a microstructure and a texture of the 400R sample measured by ACOM. Only grains elongated along RD are observed while no recrystallised grains occur in the deformed state. The grains have two different sizes of around 100 µm and more than 1000 µm measured along TD (Fig. 1(a)). This is due to the microstructure of the starting material before warm rolling [4]. The texture of the sample, shown in the $\varphi_2=45$ section of the ODF in Fig. 1, consists of a strong γ fibre component with a maximum peak of nearly 19.0 and a rotated cube component. There was no significant difference in grain structure and macroscopic texture between the 400R and the 600R samples.

Recrystallisation kinetics. Figure 2 shows a partially recrystallised microstructure of the 600R sample heat treated at 900 °C for 1 min. Inhomogeneously distributed fine recrystallised grains with almost equiaxed shape are observed. The recrystallised area fraction was 59%. The fractions were measured for all samples from areas with twice the size of the micrograph in figure 2.

Figure 3 shows change in the recrystallised area fraction with annealing at 900, 800 and 700 °C for the two rolled samples. The curves have the typical S-shape of recrystallisation and the rate increases with increasing temperature as expected. It is, however, noted from this diagram that the samples annealed at 700° and 800°C show a large difference in the recrystallisation kinetics depending on the rolling temperature while the difference becomes much smaller at 900 °C.

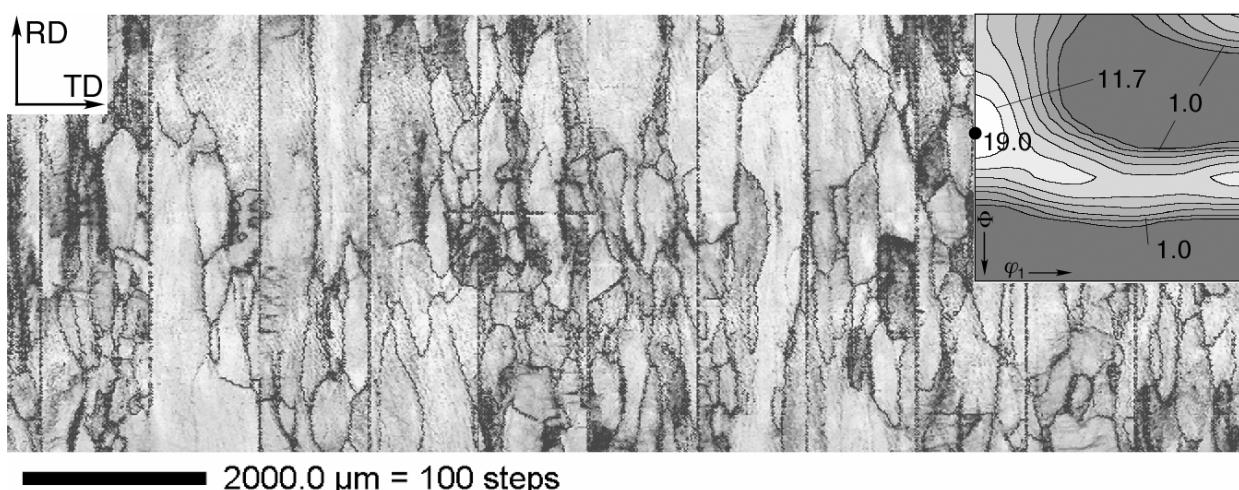


Figure 1 EBSD misorientation map taken from the normal plane and $\varphi_2=45^\circ$ section of the ODF of the alloy rolled at 400°C to a reduction of 65% (400R sample). Sharp vertical lines are due to stitching of individual measurement fields.

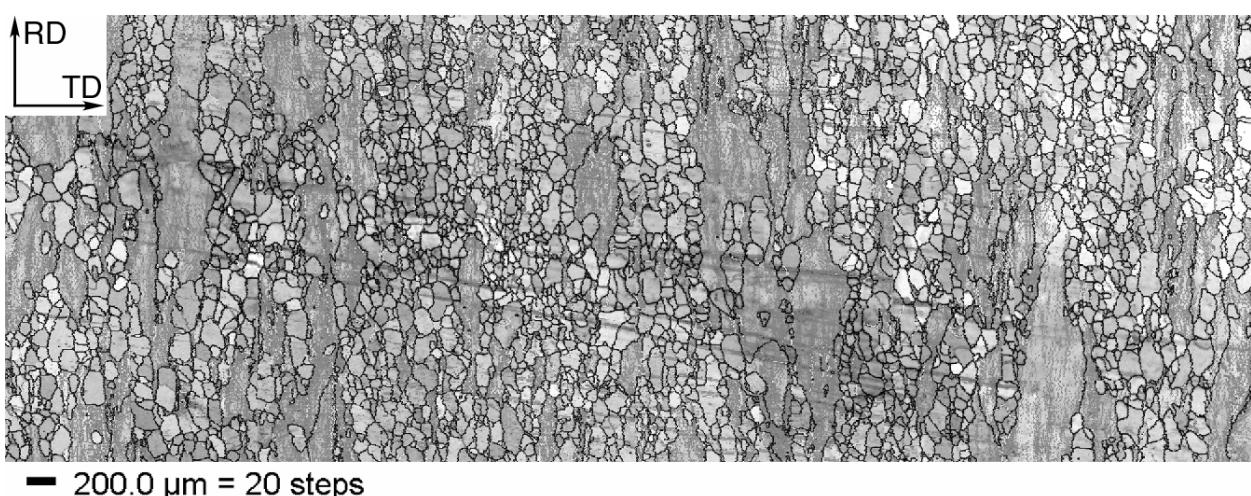


Figure 2 EBSD misorientation map of the alloy rolled at 600°C to a reduction of 73% and heat treated at 900°C for 1 min. The degree of recrystallisation is 59 %. Diagonal lines are scratches from polishing.

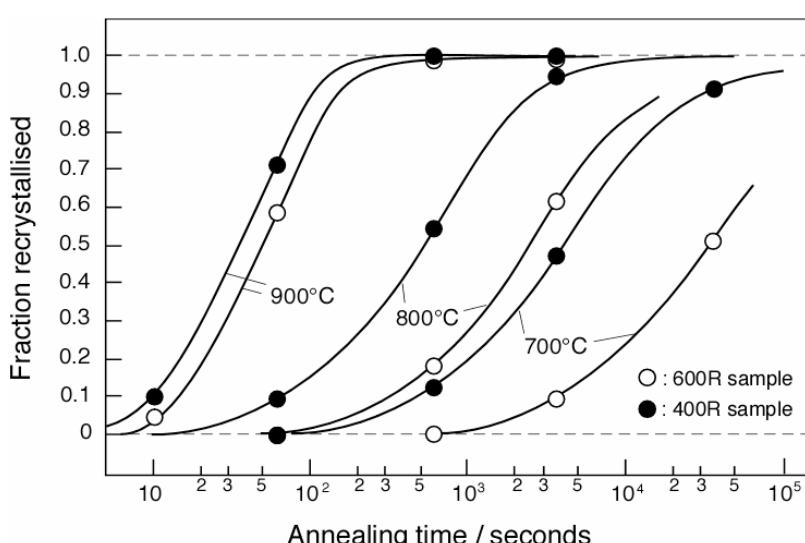


Figure 3 Change in recrystallised area fraction with annealing at 900, 800 and 700 °C for the samples rolled at 600°C to 73% (600R sample) and 400°C to 65% (400R sample).

The times for 50% recrystallisation ($t_{0.5}$) were estimated from the curves in figure 3 and plotted against reciprocal temperature as shown in figure 4. The drastic decrease in difference of the recrystallisation kinetics between the two rolled samples at 900 °C is more visible. Another remarkable feature is that the apparent activation energy in the ordered state is lower than that in the disordered state. From the slope of the data derived from the 600R sample, an activation energy of 234 kJmol⁻¹ is obtained for the ordered state, while for the disordered state the activation energy is 408 kJmol⁻¹^a. It is also noted that the difference between the activation energies for the ordered and the disordered state is much less significant in the data derived from the 400R sample. The meaning of the dashed lines is explained in discussions.

Discussions

Difference in recrystallisation kinetics between the 600R and 400R samples. It was reported that in ordered materials the degree of order decreases with deformation [2, 3]. It is furthermore expected that rolling at different temperatures leads to a different degree of order. During subsequent annealing different kinetics of ordering or disordering may therefore affect the recrystallisation kinetics of the two rolled samples. There is, however, no significant difference in the degree of order between the 600R and 400R samples. Figure 5 shows change in the degree of order with annealing at 700 °C. Values of 0.77 and 0.68 were obtained from the 600R and 400R samples, respectively. The degree of order increases by only 0.1 with a small jump during annealing. This jump may be due to the onset of the recrystallisation since it occurs after the same annealing time as the onset of recrystallisation (Fig. 3). It is therefore not reasonable to assume that the difference in the degree of order has a great influence on the difference in the kinetics of recrystallisation between the 600R and 400R samples.

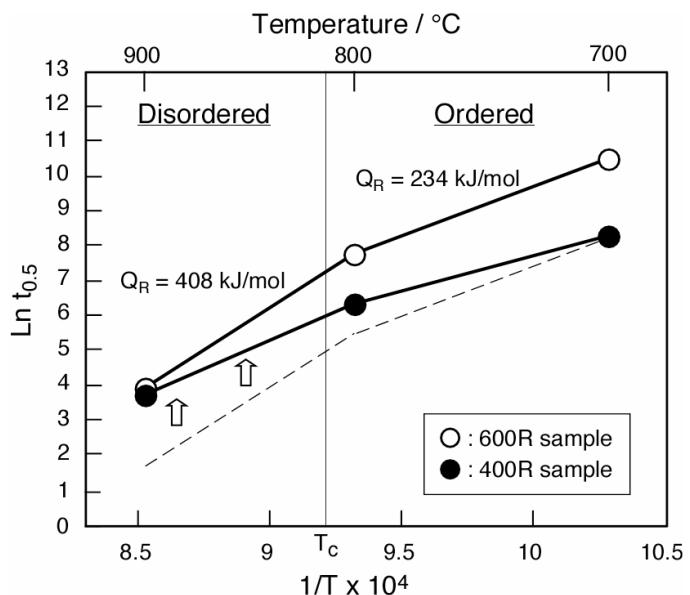


Figure 4 Arrehenius plot showing temperature dependence of the time required for 50% recrystallisation. The dashed lines correspond to the prediction of recrystallization time for the 400R sample without recovery effect.

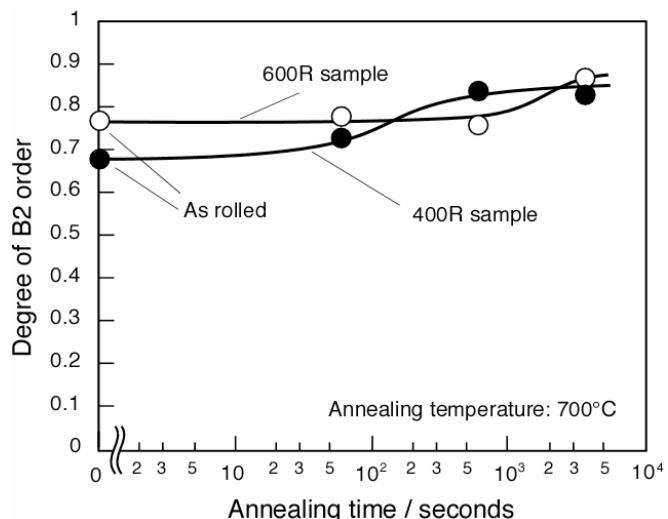


Figure 5 Degree of order of the 600R and the 400R samples with annealing at 700 °C in dependence of the annealing time

^a Since the A2-B2 transition is a second order transformation, it is expected that the data show a bend in recrystallisation kinetics around T_c . In the case of a first order transformation [6, 7], the data show a jump at T_c . Therefore, our estimation of the activation energy in the disordered state seems to be reasonable.

Another possible reason that may account for the difference is different stored energies in the two rolled samples and their different recovery rates during annealing at each temperature. Figure 6 shows the change in the half-width of the diffraction peak (400) of the two rolled samples with annealing. This figure also contains a reference value (0.33) from a completely recrystallised structure. The 400R sample has nearly a three times larger value compared to the reference, while the 600R sample's value is not as high, indicating that the 400R sample has a much larger stored energy than the 600R sample in deformed state. During annealing at 900°C the high value in the 400R sample rapidly decreases and joins the curves of the data derived from the 600R sample just after 10 seconds, while at 800 and 700 °C the values still keep higher than the 600R curves even after several minutes. These results clearly demonstrate that in the beginning of recrystallisation at 900 °C the 400R sample reduced its stored energy to almost the same level as the annealed 600R sample, while in annealing at 800 °C and 700 °C there are still differences in stored energy in the beginning of recrystallisation between the 600R and 400R samples, resulting in the drastic decrease of the difference in recrystallisation kinetics between the 600R and 400R samples at 900 °C. It is currently investigated, whether there is an effect of disordering on the kinetics of recovery.

The activation energy for recrystallisation in disordered and ordered state. The change of recrystallisation kinetics described above is reflected in a reduction of the difference between the activation energies for the ordered and the disordered state in sample 400R (Fig. 4). This is attributed to the effect of a higher recovery rate at higher temperatures in the 400R sample. Without recovery effect the arrhenius plot for sample 400R would therefore correspond to the dashed lines in figure 4. Based on this consideration, it is therefore favourable to evaluate the apparent activation energies for recrystallisation in the ordered and the disordered state from the plot for the 600R sample. It should be kept in mind, however, that also these values are still underestimated since a little recovery also occurs in this sample during annealing.

A lower activation energy in ordered state than in disordered state is also reported in the literature [6-8]. These results seem to be opposite to the fact that the activation energy of diffusion in the ordered state is higher than that in the disordered state [9]. Cahn et al. [6] for an L1₂-fcc system explained that the apparently lower activation energy in the ordered state is simply due to the reduction of driving force by recovery, which strongly influences the observed recrystallisation kinetics in the ordered state, but not in the disordered state. However, this explanation cannot be applied to this study because the recovery effect was shown to be more pronounced in the disordered state than the ordered state. Hutchinson et al. [7] concluded that the mechanism of recrystallisation in the ordered state involves migration of only low or medium angle grain boundaries with a lower activation energy than that of high angle grain boundaries, while in the disordered state the normal process of high angle migration prevails.

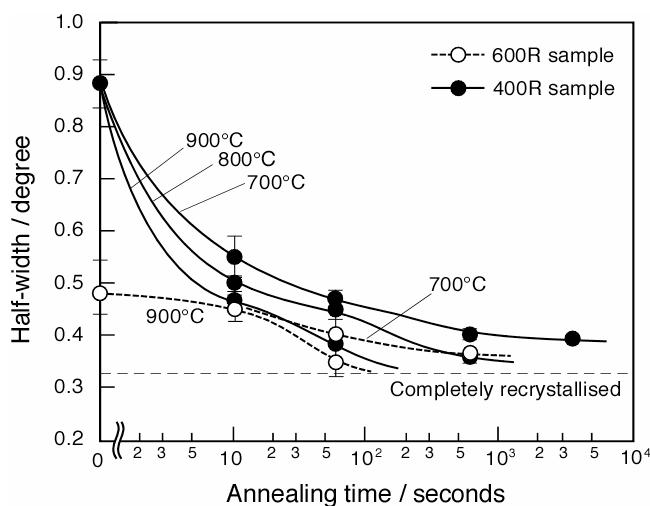


Figure 6 Half-width (FWHM) of the (400) diffraction peak of the two rolled samples with annealing at 900°C, 800°C and 700°C in dependence of annealing time.

The mechanism for the lower activation energy in the ordered state in this study has not been fully understood yet, but there is an apparent difference in recrystallisation manner between in the ordered and the disordered states, as briefly explained in the following.

Above T_c : a small grain size after 50 % recrystallisation indicates a high frequency of nucleation.

Below T_c : the larger grain size and smaller number of grains indicates a lower frequency of nucleation and grain growth with migration of high angle grain boundaries.

These results demonstrate that above T_c recrystallisation is more controlled by nucleation, while below T_c growth of the recrystallised grains is predominant. Nucleation of recrystallisation is controlled by the migration of low angle grain boundaries from pre-existing subgrains [9]. On the other hand, grain growth occurs by large angle grain boundary migration. It has been reported that the activation energy for low angle grain boundaries migration has a significantly larger value than that for high angle grain boundaries migration [10, 11]. The ratio of both values is close to the values that we determined. Thus, in the disordered state the rate-controlling nucleation mechanism would thus result in a higher activation energy for recrystallisation than that for boundary movement in the ordered state.

Summary

In the present study the kinetics of recrystallisation in A2 and B2 phase regions for a Fe₃Al based single phase alloy rolled at 400 °C and 600 °C were determined in conjunction with the kinetics of the A2-B2 ordering and recovery. The results can be summarised as follows;

1. Depending on rolling temperature there are large differences in the kinetics for recrystallisation at 800 °C and 700°C, while the difference becomes much smaller at 900 °C. These results are attributed to the high stored energy in the sample rolled at 400 °C and its high recovery rate at 900 °C, leading to a similar level in stored energy as for the sample rolled at 600 °C in the beginning of annealing.
2. Almost half the apparent activation energy in the ordered state than that in the disordered state was observed. This could be related to the difference in recrystallisation manner, i.e., above T_c recrystallisation is more controlled by nucleation, while below T_c more controlled by grain growth.

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