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The Relative Importance of Nucleation vs. Growth for Recrystallisation in Particle-containing Fe_3Al Alloys

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Abstract. Recrystallisation behavior was studied in two Fe_3Al-based alloys containing both large and fine particles with a different fine particle dispersion level using high-resolution SEM and EBSD. High misorientation of 15-30° was created around large particles after a hot rolling process in the two alloys. The kinetics of recrystallisation were, however, considerably retarded in the alloy containing dense fine particles. It was observed that the growth of subgrains created around the large particles was inhibited by the presence of the fine particles. This result clearly suggests that when the particle density (N_s) is high relative to local stored energy (E) around large particles, *nucleation can be completely hindered*. As the N_s/E level decreases, nucleation may occur and the kinetics of recrystallisation might be determined by both the nucleation rate and the growth of nuclei into a matrix with fine particles.

Introduction

We have been working on microstructure control in Fe_3Al (bcc derivative structure) based alloys by means of a thermomechanical process [1-3]. Our main purpose through the process is to obtain a fine grained and recovered structure stabilized by fine particles for improving toughness and creep resistance. The process contains two parts; the first part is grain refinement and the second the creation of deformed grains with high density of fine particles to promote extended recovery. It has been found in the first part of the process that large particles are required to refine grain size by particle stimulated nucleation (PSN) as well as to pin grain boundaries in a conventional hot rolling and annealing process [1-3]. We are, therefore, recently studying recrystallisation behavior of alloys containing both large and fine particles for the second part of the process.

Particles may affect the two stages of recrystallisation, i.e. nucleation and growth, in the following ways: (a) large particles to promote nucleation by PSN, (b) fine particles to hinder nucleation by pinning sub-boundaries and (c) fine particles to retard growth by pinning grain boundaries [4]. In alloys with bimodal particle distributions, Nes et al. [5] observed coarse recrystallised grains and explained this result in the viewpoint of *nucleation*, i.e. the Zener pinning pressure may increase the critical particle diameter for PSN, thereby hindering nucleation. Chan and Humphreys [6] found slow kinetics of recrystallisation and a microstructure consisting of large irregular grains and small island grains. These observations were discussed in terms of retardation of *growth* by the interaction of high angle grain boundaries and pinning particles.

The objective of this study is to investigate experimentally which process, formation of a nucleus with particles or its growth into a particle-containing matrix, is important for recrystallisation kinetics in Fe_3Al-based alloys containing both large and fine particles.

Experimental Procedures

Two Fe-26Al based alloys with additions of Cr, Mo and C were studied. These alloys will be designated with their Cr, Mo and C content in atomic percent as shown in all the tables in this paper. 2 kg ingots of these alloys were prepared from 3N purity iron, 4N aluminium, 3N chromium, 3N

molybdenum and 3N carbon by induction melting in an argon atmosphere. The surface of the ingot was machined and was then cut into blocks of 56 x 27 x 80 mm. After obtaining fine grains of 70-80 μm in diameter by hot rolling at 800 °C to a reduction of 65 % and an annealing heat treatment at 1000 °C for 20 min, the plates were immediately hot rolled to a reduction of 50 % in 4 passes without intermediate heat treatment. The hot rolled plates were subsequently cut into pieces of 12 x 4.7 x 10 mm and annealed at 700 °C for 1 to 60 hours in air followed by water quenching.

The transverse sections, consisting of normal direction (ND) and transverse direction (TD), were used for microstructure observations. The section was ground, mechanically polished down to 3 μm diamond paste and finished by mechanical and chemical polishing using SiO_2 polishing suspension. Microstructure was examined by high resolution scanning electron microscopy with a backscattered electron (BSE) detector and electron backscatter diffraction (EBSD) camera. Volume fraction, particle length and number density of particles were measured using image processing software.

Results and Discussion

Deformed Microstructure around Large κ Phase Particles. The two alloys studied contain large κ-Fe_3AlC ($E2_1$) phase particles after hot rolling. Measured volume fraction, particle length and interparticle spacing of the phase are given in Table 1. A similar particle dispersion level was obtained for the two alloys although the particle size is slightly larger and less dense in the alloy 0-1-1.2. A BSE image of the hot rolled alloy 2-1-1.2 is shown in Figure 1. Orientation contrast is clearly seen around an average-sized κ phase particle. The orientation profile of the matrix along the line AB with respect to the point A is shown in the right side of figure 1. The total misorientation of 26° was created in the deformation zone of which the size is about 20 μm.

Table 1 Particle dispersion parameters of large κ phase for the alloys after hot rolling

Alloy designation	Volume fraction	Particle length / μm			Interparticle spacing (λ) / μm
		Min.	Ave.	Max.	
2-1-1.2	7,6%	4,2	9,6	33,5	21,0
0-1-1.2	7,8%	4,0	12,2	59,4	24,7

$\lambda = N_s^{-1/2}$, N_s: number of particles per area.

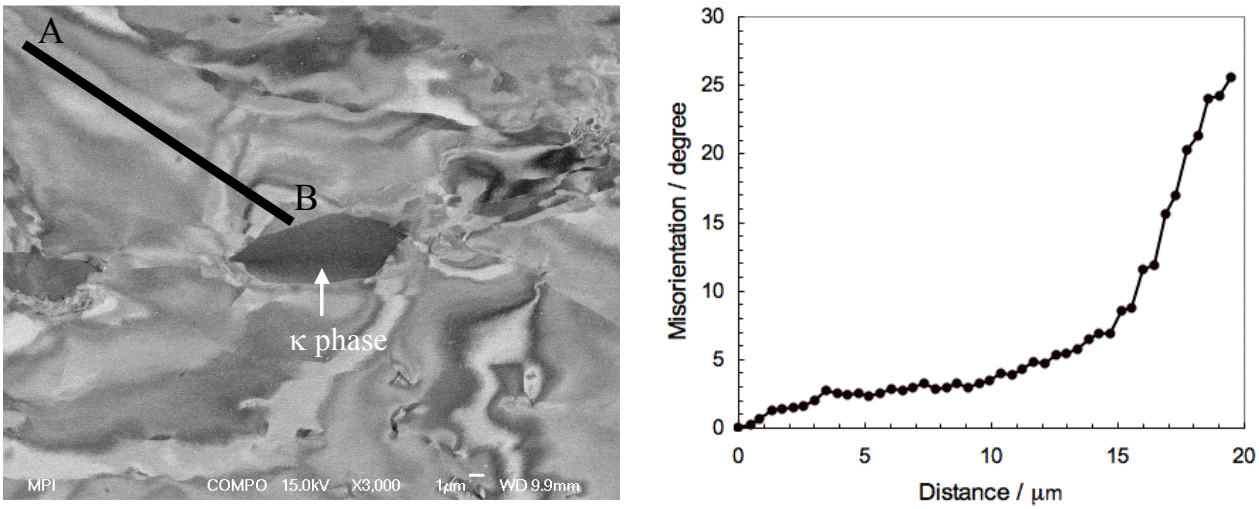

Figure 1 A backscattered electron image of the alloy 2-1-1.2 after hot rolling showing a clear orientation contrast in the matrix around large κ phase (left), and the misorientation profile of the matrix phase along the line AB (right).

Table 2 shows ten measurements for the two alloys each of the maximum total misorientation values obtained from deformation zones around average-sized κ particles. This result shows that 15-30° of total misorientation is available in a fairly large deformation zone in almost all the cases, indicating that 10 μm-sized κ particles may act as nucleation sites in a subsequent annealing process.

Table 2 The maximum misorientations obtained in deformation zones of the matrix around 10 μm-sized κ phase particles

Alloy designation	Particle number	Particle length /μm	Total misorientation / degree	Size of deformation zone / μm
2-1-1.2	1	11	26	20
	2	8	22	11
	3	13	30	12
	4	11	17	6
	5	10	17	13
	6	10	17	22
	7	8	15	12
	8	12	19	10
	9	10	27	11
	10	7	16	15
0-1-1.2	1	10	22	11
	2	8	24	12
	3	9	20	10
	4	9	32	19
	5	9	21	10
	6	10	25	21
	7	10	26	12
	8	12	24	15
	9	12	14	17
	10	13	24	17

Formation of Fine M_2C Particles during Annealing. Very fine M_2C ($B8_1$) carbide phase was precipitated during annealing in the alloy 2-1-1.2. Figure 2 shows BSE images of the alloy after annealing at 700 °C. After 1h annealing (left), fine and needle-like precipitates (M_2C) were densely formed around the κ phase with a thin particle free zone (PFZ) of 1-2 μm in width. It is noted that clear orientation contrast is still visible around the κ phase, which may allow us to determine effects of the fine particles on the nucleation process. Additional information which should be mentioned here is the κ phase shrinking during annealing since this phase is thermodynamically metastable at this temperature [7]. Original interfaces are decorated by M_2C particles as shown by arrows in the figure. With annealing particles are coarsening and the width of the PFZ decreases (Figure 2(right)). Measured particle parameters of the M_2C phase for the two alloys are given in Table 3. It can be seen that the fine dispersion level remains high up to 60 h annealing in the alloy 2-1-1.2. Although the alloy 0-1-1.2 also forms M_2C particles, the number density of the particles in this alloy is much lower than that in the alloy 2-1-1.2.

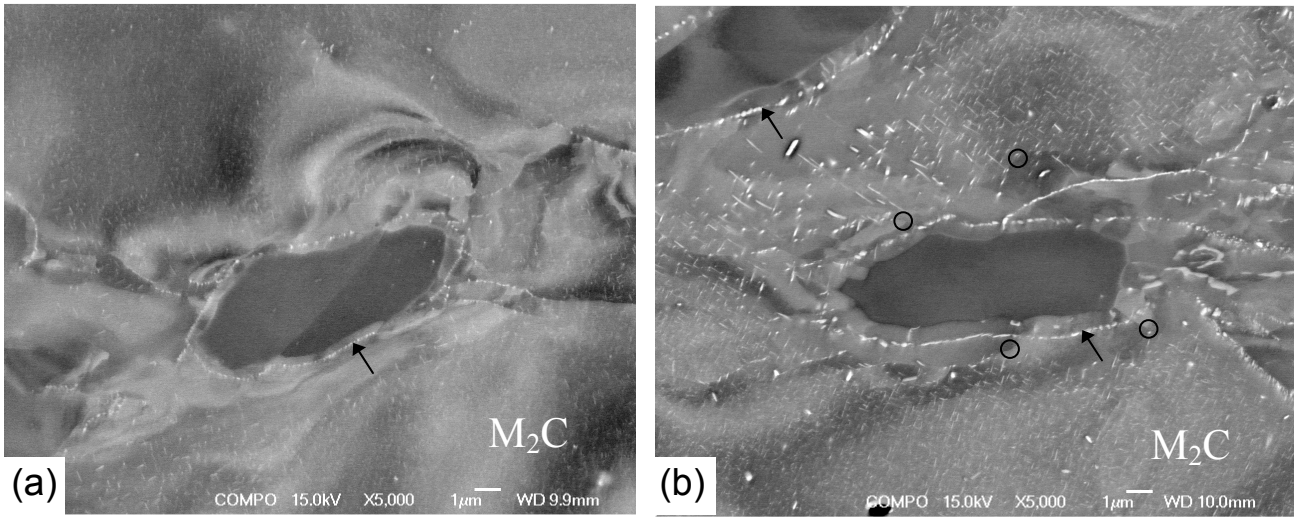

Figure 2 Backscattered electron images of the alloy 2-1-1.2 after annealing at 700 °C for 1h (a) and 24h (b). Fine M_2C particles are observed around the large κ phase with a 1-2 μm-sized PFZ.

Table 3 Particle parameters of fine M_2C phase in the alloys annealed at 700 °C

Alloy designation	Anneaing time / h	Volume fraction	Particle length / μm			Interparticle spacing (λ) / μm
			Min.	Ave.	Max.	
2-1-1.2	1*	2,5%	<0.01	0,03	0,4	0,29
	24	4,4%	<0.01	0,07	1,3	0,24
	60	4,5%	<0.01	0,11	1,3	0,37
0-1-1.2	1	0,1%	<0.01	0,08	0,2	2,0
	24	2,7%	<0.01	0,29	1,5	1,1
	60	3,3%	<0.01	0,27	2,2	1,1

$\lambda = N_s^{-1/2}$, N_s: number of particles per area.

* Very fine particles are probably not measured due to the limitation of image resolution.

Kinetics of Recrystallisation. Change in the recrystallised area fraction with annealing is shown in Figure 3 for the two alloys. It can be seen that the recrystallisation in the alloy 2-1-1.2 with densely formed M_2C particles was considerably hindered even after 60 h, whereas recrystallisation proceeds in the alloy 0-1-1.2 by 35% in fraction. In order to explain this drastic retardation of recry stalli- sation in the former alloy, local orient- tations of the matrix around the large κ phase were measured by EBSD in the annealed samples.

Figure 4 shows an EBSD map obtained from the same area as shown in Figure 2(b). High angle grain boundaries (HAGB) and low angle grain boundaries (LAGB) are drawn as black and white lines, respectively.

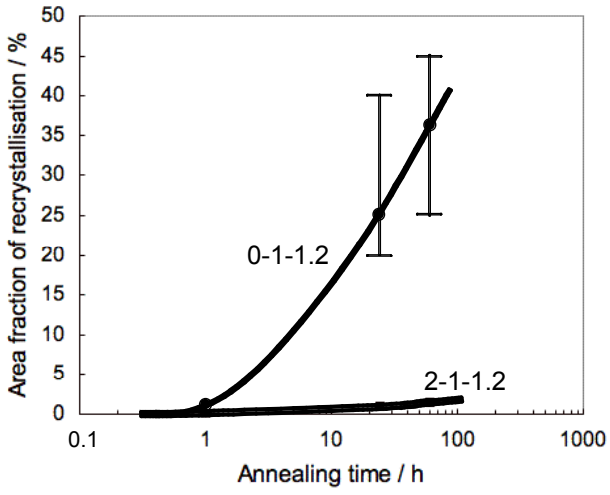

Figure 3 Change in recrystallised area fraction with annealing at 700 °C for the alloys studied.

Comparison of the two figures shows that most boundaries which are visible in the BSE (examples are indicated by circles in Figure 2(b)) are sub-boundaries. As shown in the orientation profiles of the lines 1 and 2 in figure 4 (right), there still exists orientation gradient in front of the subgrains, which indicates that the subgrains have the driving force to grow to be nuclei. It seems from these observations that the presence of the fine M_2C particles ahead of the subgrains plays an important role in inhibiting the migration of sub-boundaries.

The subgrain size and the width of PFZ as a function of annealing time is shown in Figure 5 for the alloy 2-1-1.2. These results were obtained from at least three orientation maps around 10 μm-sized κ phase for each of the three annealed samples. With annealing the subgrain size increases to about 1 μm and remains after 24h. On the other hand, the PFZ width decreases and becomes similar to that of the subgrain size. This unchanged subgrain size with annealing and its accordance with the PFZ width clearly demonstrate that the fine M_2C particles are pinning LAGB in the nucleation process of

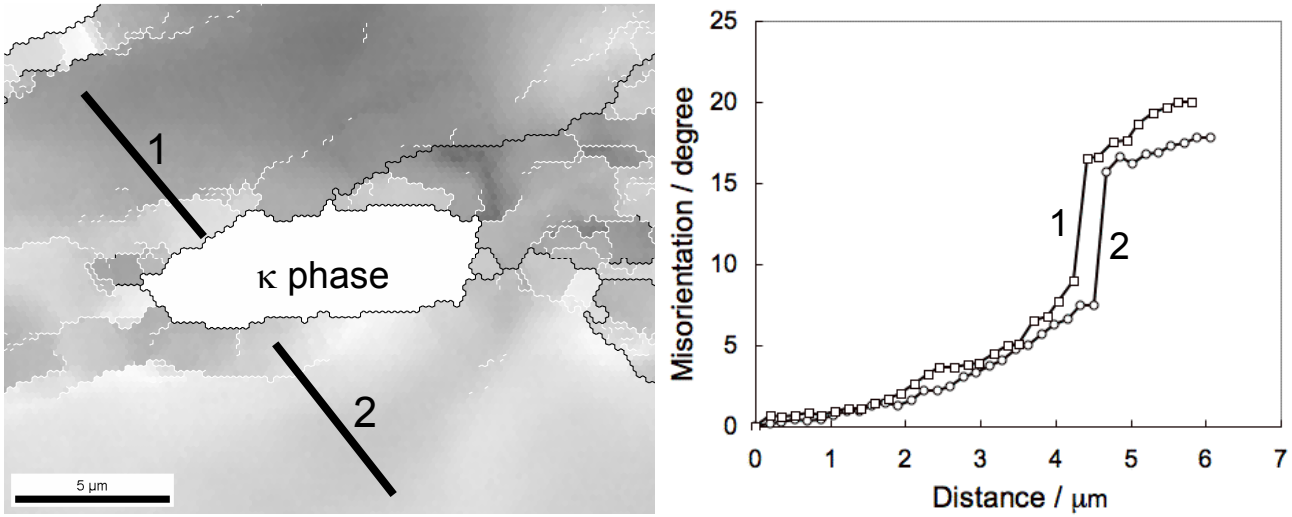

Figure 4 EBSD map showing HAGB (black) and LAGB (white) obtained from the same area as shown in Figure 2(b) (left), misorientation profiles of the matrix along the lines 1 and 2 (right).

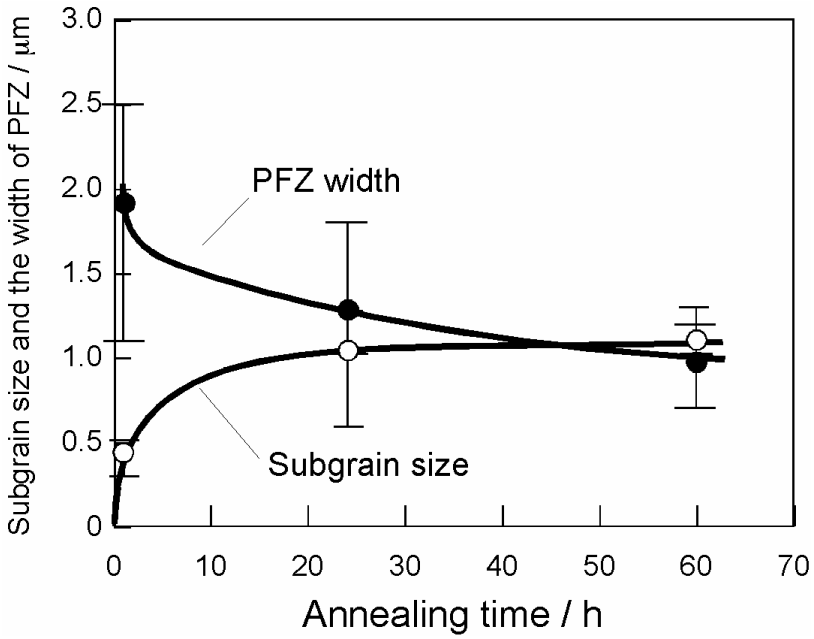

Figure 5 Change in the subgrain size and the width of particle free zone with annealing at 700 °C in the alloy 2-1-1.2.

The Relative Importance of Nucleation vs. Growth for Recrystallisation. Based on the results obtained above and knowledge from literature [4,6], it is assumed that the relative importance of nucleation vs. growth for recrystallisation depends on the fine particle dispersion condition as well as local stored energy around large particles. When the particle density (N_s) is high relative to local stored energy (E), and the N_s remains unchanged during annealing, nucleation can be completely hindered. Particle free zones around large particles should be small to obtain this case. As the particle density level (N_s/E) decreases, nucleation may occur and the kinetics of recrystallisation might be determined by both the nucleation rate and the growth of nuclei into a matrix with fine particles. We assume that the samples showing large and small grains reported in literature [6,8] belong to this case. When the N_s/E level is very low, PSN will dominate recrystallisation kinetics and fine grain sizes can be achieved [6,9].

Summary

Recrystallisation behavior was studied in two Fe_3Al-based alloys containing large and fine particles with a different fine particle dispersion level. The conclusions of this study are as follows:

(1) High misorientation of 15-30° was created around large particles after a hot rolling process in the two alloys.
(2) The kinetics of recrystallisation were, however, considerably retarded in the alloy containing densely precipitated fine particles. It was observed that the growth of subgrains created around the large particles was inhibited by the presence of the fine particles.
(3) This result clearly suggests that when the particle density (N_s) is high relative to local stored energy (E) around large particles, nucleation can be completely hindered. As the N_s/E level decreases, nucleation may occur and the kinetics of recrystallisation might be determined by both the nucleation rate and the growth of nuclei into a matrix with fine particles.

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Fundamentals of Deformation and Annealing
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