Texture Development during Dynamic Recrystallization in Hot-deformed Fe–40at%Al Alloy

T. SAKATA, H. KOHMA, H. YASUDA and Y. UMAKOSHI

Department of Materials Science and Engineering & Frontier Research Center, Graduate School of Engineering, Osaka University, Yamada-oka, Suita, Osaka 565-0871 Japan.

(Received on March 19, 2002; accepted in final form on May 14, 2002)

1. Introduction

FeAl with the B2 structure is expected to be a potential substitute for stainless steels operating at intermediate temperatures because of its low material cost, low density, and excellent sulfidation and oxidation resistance.1,2) The obstacles to its application are limited ductility and low fracture toughness at ambient temperature due to dynamic environmental embrittlement, particularly grain boundary embrittlement.3,4) Mechanical properties of some compounds are known to depend on the microstructure and crystal orientation distribution.5–8) Development of the process to obtain the favorable microstructure and crystal orientation distribution is necessary. The microstructure and crystal orientation distribution in hot-compressed Fe–40at%Al alloy were investigated using an electron back-scatter diffraction pattern technique. After the hot deformation, grain boundaries became more serrated with increasing tested temperature or decreasing strain rate, i.e., decreasing the Zener–Hollomon parameter (Z). Equiaxed fine grains surrounded by high angle boundaries were homogeneously formed due to dynamic recrystallization accompanied by serrated grain boundaries during hot deformation at lower Z. Moreover, strong (111) fiber texture parallel to the compressive axis developed at larger strains because of the lattice rotation during hot deformation, which was good agreement with the stable orientation calculated by Taylor model. The microstructural factors which established the mechanism of texture evolution during dynamic recrystallization were examined focusing on the Z parameter.

KEY WORDS: iron aluminides (based on FeAl); hot deformation; dynamic recrystallization; lattice rotation; texture; Zener–Hollomon parameter.

2. Experimental Procedure

Master ingots of Fe–Al alloy containing 40 at% Al (nominal composition) were prepared by melting high purity Fe (99.9%) and Al (99.99%) in a plasma arc furnace. The actual composition of the ingot was determined to be Fe–39.5at%Al by an electron-probe microanalysis (EPMA). Cylindrical specimens with 8 mm in diameter and 12 mm in height were cut from the ingots. The hot-compression was applied for the specimens to control the microstructure using a thermomechanical-processing simulator (Fuji Electronic Industrial Co., Ltd. Thermeastor-Z); the cylindrical specimens were first annealed in an Ar gas atmosphere at 1273 K for 0.5 h for homogenization and subsequently compressed at strain rates (\(\dot{\varepsilon}\)) of \(1.0 \times 10^{-3}\)–\(1.0 \times 10^{-1}\) s\(^{-1}\) to true strains (\(\varepsilon\)) of 0.4, 0.9 and 1.6 at temperatures between 1073 and 1273 K, and then finally quenched by Ar gas flow. The strain rate was controlled to be constant during compression. The initial grain size just
800 to develop an orientation map over an area of 80/11003 behavior suggests that dynamic recovery or dynamic recrystal-
temperatures or lower strain rates. The work softening be-
in the flow can be seen in the stress–strain curves at higher
0.7. A steady-state flow accompanied by a slight decrease
creases with increasing plastic strain up to true strain of
ing strain rate (Fig. 1(b)) at 1 273 K. The flow stress de-

before the hot compression is approximately 500 μm. On
the other hand, the coarse grained specimens with more
than 1 mm grain diameter, homogenized at 1 373 K for 24 h
before hot compression were also used for the test to examine
the characteristics of grain-boundary serration.

The hot compressed specimens were cut in the normal
direction at the center of the cross-section parallel to the
compressive axis by spark machining. The specimen surface
was electrolytically polished in a methanol-based solution
containing 10% perchloric acid after mechanical poli-
ishing and then microstructurally observed by an optical
microscopy. Thin foils for transmission electron microscop-
ic (TEM) observation, cut parallel to the compressive axis
were perforated by twin-jet method. Texture analysis and
crystallographic observation were carried out using an elec-
tron back-scatter diffraction pattern (EBSP) technique. The
electron beam was automatically moved in 1–10 μm steps
before hot compression is approximately 500 μm. On
the other hand, the coarse grained specimens with more
than 1 mm grain diameter, homogenized at 1 373 K for 24 h
before hot compression were also used for the test to examine
the characteristics of grain-boundary serration.

The hot compressed specimens were cut in the normal
direction at the center of the cross-section parallel to the
compressive axis by spark machining. The specimen surface
was electrolytically polished in a methanol-based solution
containing 10% perchloric acid after mechanical poli-
ishing and then microstructurally observed by an optical
microscopy. Thin foils for transmission electron microscop-
ic (TEM) observation, cut parallel to the compressive axis
were perforated by twin-jet method. Texture analysis and
crystallographic observation were carried out using an elec-
tron back-scatter diffraction pattern (EBSP) technique. The
electron beam was automatically moved in 1–10 μm steps
before hot compression is approximately 500 μm. On
the other hand, the coarse grained specimens with more
than 1 mm grain diameter, homogenized at 1 373 K for 24 h
before hot compression were also used for the test to examine
the characteristics of grain-boundary serration.

The hot compressed specimens were cut in the normal
direction at the center of the cross-section parallel to the
compressive axis by spark machining. The specimen surface
was electrolytically polished in a methanol-based solution
containing 10% perchloric acid after mechanical poli-
ishing and then microstructurally observed by an optical
microscopy. Thin foils for transmission electron microscop-
ic (TEM) observation, cut parallel to the compressive axis
were perforated by twin-jet method. Texture analysis and

stress gradually increases with further compression because
of the formation of dead metal zones in the high strain re-
gions. The Zener–Hollomon parameter (Z) defined by Eq.
(1) is known to be useful for understanding hot working
process.

\[ Z = \dot{\varepsilon} \exp(\frac{Q}{RT}) \] \hspace{1cm} (1)

where \( \dot{\varepsilon} \) is strain rate, \( R \) gas constant, \( T \) absolute temperature
and \( Q \) activation energy. The \( Q \) value was determined
to be 306 kJ mol\(^{-1}\) from the flow stress at a strain of \( \varepsilon=0.3 \)
at various testing conditions. The testing conditions without
a steady-state flow was excluded from the calculation of the
\( Q \) value. The values of the \( Z \) parameter at the different con-
ditions are summarized in Table 1. The flow stress decreases
with decreasing \( Z \). Moreover, the steady-state flow can be
observed at lower \( Z \), i.e., at logarithmic \( Z \) values of 9.99
and 9.56.

Figure 2 shows microstructures in Fe–40at%Al alloy
compressed to \( \varepsilon=1.6 \) at various levels of the \( Z \) parameter.
At high \( Z \) value of \( \log Z=11.90–10.56 \), original grains be-
come flattened and the grain boundaries are frequently ser-
rated, as shown in Figs. 2(a), 2(b), 2(c) and 2(d). The grain
boundaries become more serrated with decreasing \( Z \).
Moreover, small grains are sometimes pinched-off from the
serrated parts of initial grains as indicated by arrows.
The morphology of the grain structures is similar to the one
often observed during dynamic recrystallization. Moreover,
equiaxed grains are homogeneously distributed at lower \( Z \)
of \( \log Z=9.99 \) or 9.56, as shown in Fig. 2(e) or 2(f), al-
though their initial grain size was about 500 μm. Although
the photograph is not shown here, numerous dislocations
and subgrains were observed in each grain by TEM obser-
vation. This strongly suggests that dynamic recrystalliza-
tion accompanied by the grain-boundary serration occurs in
hot-compressed Fe–40at%Al alloy.

Figure 3 shows EBSP analysis of the specimens com-
pressed to various strains at \( \log Z=9.56 \) (at 1 273 K and
\( \dot{\varepsilon}=1.0 \times 10^{-3} \) s\(^{-1}\)). Thin and bold lines in orientation imag-
ing micrographs (OIM) represent grain boundaries with
misorientation angle (\( \Delta \theta \))=1°–15° and more than 15°, re-
spectively. In the present study, a grain surrounded by
boundaries with \( \Delta \theta=1°–15° \) is regarded as a subgrain. At
\( \varepsilon=0.4 \), the shape of initial grains changes to a pancake-like
morphology with serrated grain boundaries and numerous
subgrains are formed in the original grains, as shown in
Fig. 3(a). In contrast, the equiaxed grains with about 135
μm in diameter surrounded by high angle boundaries
(\( \Delta \theta>15° \)) are homogeneously formed at strains more than
\( \varepsilon=0.9 \) and numerous subgrains also exist, as shown in Figs.
The refinement of grains with subgrains and high frequency of high angle boundaries strongly suggest that the dynamic recrystallization occurs during hot deformation.

Figure 4 shows the inverse pole figures on the compressive plane at log $Z=9.56$ (at $1273$ K and $\dot{\varepsilon}=1.0 \times 10^{-3}$ s$^{-1}$). The grains are randomly oriented at $\varepsilon=0.4$ (Fig. 4(a)), while (111) fiber texture along the compressive axis is well developed at larger strains of $\varepsilon=0.9$ and 1.6 (Figs. 4(b) and 4(c)). The fractions of (111) oriented grains at a tolerance angle of 15° over the whole area of EBSP measurement at $\varepsilon=0.4$, 0.9 and 1.6 were nearly 48%, 58% and 75%, respectively. Thus, the texture monotonously develops with increasing strain, but no distinct changes in both size and shape of grains were observed at strains more than $\varepsilon=0.9$. This implies that the texture evolution proceeds because of the lattice rotation by slip deformation even after the whole area of the specimens was replaced with dynamically recrystallized grains.

The formation process of new grains under two different conditions was examined using an EBSP technique. Examples are given in Fig. 5. Since the specimens were homogenized at 1373 K for 24 h before hot deformation, the mean grain size is more than 1 mm. The coarse grain is favorable in order to examine the serrated grain boundaries. At high $Z$ (at log $Z=11.90$), the OIM map shows that the misorientation angle between the grains A with colored dark gray and A' with light gray was small less than 10°, as shown in Fig. 5(a). The grain boundaries are frequently serrated and the grains A' are pinched-off from the serrated parts of the initial grain A. In contrast, at low $Z$ (at log $Z=9.56$) the grains A, B and C represented by the colored light gray, dark gray and white, respectively are surrounded by high angle boundaries (D$\theta>15°$), as shown in Fig. 5(b). The serrated grain boundaries of the grains A and B are impinged each other so that the grain C is subdivided into two
grains. Since the separated grains are surrounded by high angle boundaries and contain subgrain structure during hot deformation, they seem to be dynamically recrystallized new grains.

3.2. Microstructural Factors in Hot-deformed Fe–40at%Al Alloy

To clarify the formation process of texture during dynamic recrystallization in hot-compressed Fe–40at%Al alloy, the change in the microstructure was examined taking into account the Zener–Hollomon parameter.

The sizes of dynamically recrystallized grains or subgrains at $\varepsilon=1.6$ are respectively plotted against the $Z$ parameter in Fig. 6. The data were obtained by EBSP analysis. The double-logarithmic plot showed the linear relationship between the $Z$ parameter and the sizes of dynamically recrystallized grains or subgrains as some researchers have reported, but the single-logarithmic plot of the $Z$ parameter in Fig. 6 will provide valuable information on the predominant mechanism of dynamic recrystallization. Both sizes abruptly increase around $\log Z=10.6$ with decreasing the $Z$ parameter. This suggests that predominant mechanism generating new grains changes depending on $Z$. The size of dynamically recrystallized grains strongly depends on that of subgrains at any $Z$ deformations since dislocation substructures such as subgrain structure during hot deformation help the grain boundary migration for generating new grains. Thus, the formation of subgrain structure is closely related to the occurrence of dynamic recrystallization accompanied by the grain-boundary serration.

The change of the fraction of dynamically recrystallized grains to total grains at $\varepsilon=1.6$ is given as a function of the $Z$ parameter in Fig. 7. The dynamically recrystallized structure well develops with decreasing $Z$. For instance, the fraction of new grain is more than 80% at $\log Z=9.56$. Furthermore, equiaxed fine grains were homogeneously formed throughout the specimens at lower $Z$. Therefore, the hot deformation at lower $Z$ is effective in controlling the homogeneously distributed fine grain structure due to the dy-
namic recrystallization.

The fraction of (111) oriented grains to total grains at $\varepsilon=1.6$ is also plotted against the $Z$ parameter in Fig. 8. About 80% of total grains is oriented (111) direction at any $Z$ values, though the fraction of (111) oriented grains becomes somewhat lower at higher $Z$. This suggests that development of the strong (111) fiber texture does not result from the dynamic recrystallization and it is due to the lattice rotation by slip during hot compression.

4. Discussion

4.1. Occurrence of Dynamic Recrystallization in Hot-deformed B2-type FeAl

Most of metals and alloys are known to be classified into two types of restoration process during hot deformation, dynamic recrystallization and dynamic recovery. In order to strictly prove the occurrence of dynamic recrystallization, dislocation substructure in new grains surrounded by high angle boundaries needs to be formed during hot deformation.\(^{21}\) In this paper, new grains surrounded by high angle boundaries and subgrain structure were well observed during hot deformation in Fe–40at%Al alloy. Therefore, work softening is induced by dynamic recrystallization during hot deformation in B2 FeAl.

In the B2-type intermetallic compound FeAl, the serration of grain boundaries due to migration resulted in the dynamic recrystallization during hot deformation. The predominant mechanism accompanied by the grain-boundary serration varied depending on the tested conditions. At higher $Z$, small new grains were pinched-off from the serrated parts of initial grains and expanded into the neighboring grain flattened during hot compression. This formation process of new grains is called bulging mechanism.\(^{10,13}\) The new grains generated by this process appeared in the localized area in the vicinity of grain boundaries resulting in an inhomogeneous microstructural change. In contrast, grains subdivided by serrated grain boundaries during hot deformation resulted in the fine grain structure at lower $Z$.

This is so-called “geometric dynamic recrystallization” as reported by McQueen et al.\(^{22}\) When subgrain boundaries interact with existing grain boundaries, some parts of the grain boundaries are serrated. If grains become thin and the size approaches the height of the serrated parts of the boundaries, the serrated parts impinge on the next grain and a new grain boundary surrounded by high angle boundaries is geometrically formed. Therefore, the development of the recrystallized grains is closely related to strain and subgrain size, and the geometry of dynamic recrystallization is shown in Fig. 9. Two neighboring serrated grain boundaries impinge on each other resulting in new grains, as shown in Fig. 9(a) (Pattern 1), while the serrated grain boundary on one side of a grain impinges on a neighboring grain, as shown in Fig. 9(b) (Pattern 2). The grain-boundary serration of Pattern 2 should be bigger than that for Pattern 1.

The critical total strain and for Pattern 1 and 2, respectively expressed as

\[ \varepsilon_1 = \ln(d/2\lambda_1) \] \hspace{1cm} (2)

\[ \varepsilon_2 = \ln(d/\lambda_2) \] \hspace{1cm} (3)

where $\lambda_1$ and $\lambda_2$ are the subgrain size for Pattern 1 and 2 around the serrated grain boundaries and $d$ is the initial grain size before hot deformation. In the present work, the critical subgrain size $\lambda_1$ and $\lambda_2$ are roughly calculated to be 51 $\mu$m and 102 $\mu$m at $\varepsilon=1.6$ and $d=508$ $\mu$m. If actual subgrain size under an adequate condition exceed the calculated $\lambda$, the geometric dynamic recrystallization occurs more easily. At lower $Z$ of log $Z=9.99$ and 9.56, the actual subgrain size in Fig. 6 becomes larger than $\lambda_1$ and $\lambda_2$. Therefore, the occurrence of geometric dynamic recrystallization at low $Z$ induces homogeneously distributed equiaxed fine grains. However, the geometric dynamic recrystallization may occur even at relatively high $Z$ around log $Z=10.6$, where the transition between the two mechanisms accompanied by the grain-boundary serration takes place, since the subgrain sizes at log $Z=10.63$ and 10.56 shown in Fig. 6 nearly satisfy the condition for Pattern 1. In addition, the geometric dynamic recrystallization seems to
result in the steady-state flow accompanied by a slight decrease in the flow in the stress-strain curves at lower $Z$, as also reported in hot-deformed B2-type $\beta$-NiAl.\(^9\)

At lower $Z$, the formation of microstructure due to the geometric dynamic recrystallization was nearly completed at $\varepsilon = 0.9$ (Figs. 3 and 5); thereafter the abrupt change in the microstructure formed by geometric dynamic recrystallization hardly occurred in compressive deformation to higher strain $\varepsilon = 1.6$ (Fig. 3). Thus, the microstructure formed by geometric dynamic recrystallization seems to be maintained in the high strain region, but the residual initial grains partly remained and the compressed microstructure was not thoroughly occupied by the dynamically recrystallized grains even under the steady-state flow condition (Fig. 7). This implies that the original grains were coarse and the shear deformation due to the formation of dead metal zones at higher strains might influence the microstructure composed of dynamic recrystallized grains, though the observation was done at the center of specimen.

4.2. Development of Texture during Dynamic Recrystallization in Hot-deformed B2-type FeAl

In hot-deformed Fe–40at%Al alloy, the $\{111\}$ fiber texture strongly developed at larger strains during dynamic recrystallization even if the predominant mechanism of dynamic recrystallization changed depending on the tested conditions. As the new grain generated by the dynamic recrystallization accompanied by the grain-boundary serration inherits the orientation of the initial grains, dynamic recrystallization does not directly result in the texture formation due to lattice rotation,\(^{17}\) which may lead to the formation of $\{111\}$ fiber texture. The crystal rotation can be predicted by numerical calculation. In the present paper, the stable orientation caused by slip deformation in B2-type FeAl was calculated using van Houtte’s algorithm.\(^{23-26}\) Two different operative slip systems of $\{110\}\langle 100 \rangle$ and $\{100\}\langle 100 \rangle$ at higher temperatures in this material were taken into account in the calculation, though slip by $\{111\}$ dislocations is known to be activated at lower temperatures less than around 1 023 K in Fe–40at%Al alloy.\(^{27,28}\)

5. Conclusions

Evolution of microstructure and preferential orientation distribution in hot-compressed Fe–40at%Al alloy was investigated focusing on the formation process of texture during dynamic recrystallization, and the following conclusions were obtained;
(1) The serration of grain boundaries becomes more irregular and frequent with increasing tested temperature and/or decreasing strain rate, i.e., decreasing the Zener–Hollomon parameter ($Z$). The serration mechanism due to grain boundary migration results in the dynamic recrystallization during hot deformation.

(2) The dynamic recrystallization accompanied by the bulging of grain boundaries occurs at high $Z$, inducing the formation of small new grains pinched-off from the serrated parts of initial grains. In contrast, the equiaxed fine grains surrounded by high angle boundaries are homogeneously formed due to the geometric dynamic recrystallization at low $Z$.

(3) The $(111)$ fiber texture along the compressive axis strongly develops at larger strains under any $Z$ values. The lattice rotation by slip deformation is responsible for the formation of the texture. The texture is in good agreement with the stable orientation calculated by Taylor model.

(4) The sizes of dynamically recrystallized grains or subgrains, and the fractions of dynamically recrystallized grains or $(111)$ oriented grains are closely correlated with the $Z$ parameter. These microstructural factors give information to determine the mechanism of texture evolution during dynamic recrystallization.

Acknowledgements

This work was supported by a Grant-in-Aid for Scientific Research on Priority Area B, “Harmonic Material Design of Multi-Functional Composites” from the Japanese Ministry of Education, Sports and Culture, Science and Technology. T. Sakata would like to thank Japan Society for Promotion of Science (JSPS) for his research fellowship.

REFERENCES

1) C. G. McKamey, J. H. DeVan, P. F. Tortorelli and V. K. Sikka: J. Mater. Res., 6 (1991), 1779.
2) N. Stoloff and C. T. Liu: Intermetallics, 2 (1994), 75.
3) C. T. Liu, E. H. Lee and C. G. McKamey: Scr. Metall., 23 (1989), 875.
4) C. T. Liu and E. P. George: Scr. Metall. Mater., 24 (1990), 1285.
5) Y.-W. Kim: Acta Mater., 40 (1992), 1121.
6) D. G. Morris, S. Gunther and C. Briguet: Scr. Mater., 37 (1997), 71.
7) M. Yamaguchi, D. R. Johnson, H. N. Lee and H. Inui: Intermetallics, 8 (2000), 511.
8) Y. D. Huang, W. Y. Yang, G. L. Chen and Z. Q. Sun: Intermetallics, 9 (2001), 331.
9) T. Sakata, H. Yasuda and Y. Umakoshi: Acta Mater., 49 (2001), 4231.
10) T. Maki and I. Tamura: Tetsu-to-Hagané, 70 (1984), 2073.
11) R. A. P. Djaja and J. J. Jonas: Metall. Trans., 4 (1973), 621.
12) T. Sakai, M. G. Akben and J. J. Jonas: Acta Metall., 31 (1983), 631.
13) H. Fukutomi, K. Aoki, N. Nomoto, S. Ikeda and C. Hartig: Mater. Trans., JIM, 35 (1994), 794.
14) D. Ponge, E. Brugner and G. Gottstein: Proc. ICSMA, 10 (1994), 725.
15) H. Fukutomi, C. Hartig and H. Mecking: Z. Metallkd., 81 (1990), 572.
16) C. Hartig, H. Fukutomi, H. Mecking and K. Aoki: ISIJ Int., 33 (1993), 313.
17) H. Fukutomi, K. Aoki, S. Takagi, M. Nobuki, H. Mecking and T. Kamijo: Intermetallics, 2 (1994), 37.
18) K. E. Harris, F. Ebrahimi and H. Garmestani: Mater. Sci. Eng., A247 (1998), 187.
19) W. Skrotzki, M. Lemke, C.-G. Oertel and R. Tamm: Mater. Sci. Eng., A234–236 (1997), 739.
20) S. A. Scheff, J. J. Stout and M. A. Crimp: Scr. Metall. Mater., 32 (1995), 975.
21) N. Tsuji, Y. Matsubara, Y. Saito and T. Maki: J. Jpn. Inst. Met., 62 (1998), 967.
22) J. K. Solberg, H. J. McQueen, N. Ryum and E. Nes: Philos. Mag., 60 (1989), 447.
23) P Van Houtte and E. Aernoudt: Z. Metallkd., 66 (1975), 202.
24) P Van Houtte and E. Aernoudt: Z. Metallkd., 66 (1975), 303.
25) G. Y. Chin and W. L. Mammel: Trans. Metall. Soc. AIME, 239 (1967), 1400.
26) U. F. Kocks and H. Chandra: Acta Metall., 30 (1982), 695.
27) M. G. Mendiratta, N. K. Kim and H. A. Lipsitt: Metall. Mater. Trans. A, 15 (1984), 395.
28) P. R. Munroe and I. Baker: J. Mater. Sci., 24 (1989), 4246.
29) P. Van Houtte: Textures Microstruct., 8–9 (1988), 313.