Magnetic observation of deformation substructure in cyclically deformed Fe₃Al single crystals

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Abstract

Fe–28.0 at% Al single crystals with the D₀₃ structure were cyclically deformed in tension-compression mode at constant total-strain amplitudes (εₜ). At and less than εₜ = 0.25%, the maximum stress increased rapidly with increasing number of cycles to 20, then remained constant during further cycling. In contrast, small cyclic softening appeared at and more than εₜ = 0.30% between the initial and the final stages of cyclic hardening. The deformation substructure in cyclically deformed Fe₃Al single crystals was observed by both transmission electron microscopy (TEM) and magnetic technique. From TEM observation, pairs of superpartial dislocations with Burgers vector (b) of 1/4[111] moved dragging a next-nearest-neighbour antiphase boundary (NNN-APB) in the cyclic softening stage, though four superpartials generally moved in a group in the initial hardening stage. The change in dislocation configuration from four- to two-coupled superpartials may ease the cross-slip event resulting in the dislocation rearrangement and the cyclic softening. The magnetic properties of cyclically deformed Fe₃Al single crystals were examined by a vibrating sample magnetometer. In particular, magnetic anisotropy in the primary slip plane was evaluated by measuring high-field susceptibility in the approach to magnetic saturation at different directions of magnetic field. The cyclically deformed crystals exhibited strong magnetic anisotropy due to atomic rearrangement near NNN-APB and internal strain around screw dislocations. Two types of magnetic anisotropy were separated from each other by the Fourier transformation. In the cyclic softening stage, the amplitude of NNN-APB-dependent anisotropy rose abruptly between 10² and 10³ cycles while that due to screw dislocations remained constant during fatigue. This suggested that a disordering of D₀₃ phase due to fatigue did not occur in the softening stage. The maximum stress of Fe₃Al single crystals exceeded the required for superpartial pairs to drag NNN-APB, resulting in the cyclic softening.

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1. Introduction

Fe₃Al with the D₀₃ structure has been expected as a high temperature structural material because of its yield stress anomaly and high corrosion resistance in oxidizing and sulfidizing environments [1,2]. In earlier and recent reports, a giant pseudoelasticity was observed to occur in Fe₃Al single crystals [3–6]; the crystals have also attracted attention as a functional material. Although, the positive temperature dependence of yield stress has been extensively examined [7–10], there is little information on the fatigue behaviour [11], and for practical application, not only monotonic deformation behaviour but the fatigue behaviour should be investigated. It is also well known that the Burgers vector (b) of a superdislocation in the D₀₃ lattice is [111] and the dislocation is dissociated into four superpartials bound by two types of antiphase boundary (APB): nearest-neighbour (NN) APB and next-nearest-neighbour (NNN) APB [12]. The dislocation configuration in Fe₃Al would make the fatigue behaviour complicated.

In general, a peculiar dislocation substructure such as vein and ladder structures is formed in fatigued materials and is closely related to the fatigue behaviour [13]. Thus, clarification of the fatigue mechanism of crystalline materials requires observation of the dislocation
substructure. Since highly dense dislocations are generally introduced in cyclically deformed crystals, however, an exact evaluation of dislocation substructure is difficult by the conventional transmission electron microscopy (TEM) technique due to an overlap of defect images. It is known that magnetic properties of ferromagnetic materials strongly correlate with the deformation substructure composed of certain kinds of lattice defects [14–21]. Type and density of lattice defects can be quantitatively evaluated by means of the magnetic technique no matter how many dislocations exist in the crystals which makes this a powerful method for examining the deformation substructure in fatigued materials. In ferromagnetic intermetallic compounds, two types of magnetic anisotropy are sometimes induced by deformation. One is due to atomic rearrangement near the APB [14–16], the other is due to internal strain around dislocations [17–21]. In our previous papers, we reported the magnetic anisotropy in cyclically deformed Ni3Fe single crystals [22,23]. Ni3Fe single crystals exhibited cyclic softening during fatigue [24,25]. By analysing fatigue-induced magnetic anisotropy, the type and density of APB and dislocations could be successfully evaluated. At the early stage of fatigue, magnetic anisotropy due to APB was dominant, while dislocation-dependent anisotropy became remarkable at the late stage of fatigue [22,23]. It was therefore concluded that the cyclic softening in Ni3Fe single crystals resulted from disordering. Since Fe3Al also demonstrates strong magnetic anisotropy by deformation [14–16], the magnetic measurement contributes to the manifestation of the fatigue mechanism. In this study, fatigue behaviour of Fe3Al single crystals was examined focusing on the magnetic anisotropy induced by cyclic deformation.

2. Experimental procedure

Master ingots of Fe–28.0 at% Al alloy were prepared by a plasma arc furnace. Fe3Al single crystals were grown by a floating zone method at a growth rate of 5 mm/h. The crystals were annealed at 1373 K for 48 h for homogenisation and then at 773 K for 100 h for D03 ordering. After the heat treatment, the degree of D03 order was 0.8. Fatigue specimens with [T49] loading axis where the Schmid factor for (T01)[111] primary slip system is 0.5 were cut from the single crystals by spark machining. Total-strain controlled fatigue tests were performed in tension/compression at constant total-strain amplitudes (εt) between 0.20 and 0.50% in air at room temperature. Slip markings on the surface of fatigue specimens were observed with an optical microscope, and deformation substructure of the fatigued samples was observed by a TEM. After the cyclic deformation, cylindrical discs parallel to (T01) primary slip plane were cut from the fatigued specimens with a form of 2.5 mm in diameter and 0.8 mm in thickness.

The magnetisation process of these discs was measured by a vibrating sample magnetometer (VSM) at room temperature. An external magnetic field of 8.0 × 105 Am⁻¹ was applied in the direction with an angle φ to the primary Burgers vector (b) of [111], as shown in Fig. 1(a). The high-field susceptibility in the approach to saturation was calculated at an effective field of 4.0 × 10⁵ A/m (Fig. 1(b)).

3. Results and discussion

3.1. Fatigue behaviour of Fe3Al single crystals

Fig. 2 shows cyclic hardening curves of Fe–28.0 at% Al single crystals cyclically deformed at different εt. At and less than εt = 0.25%, the maximum stresses (σmax) increase rapidly with increasing number of cycles (N) up to 20 followed by a stress saturation. Moreover, cyclic hardening at the initial stage of fatigue becomes strong as εt rises. In contrast, weak cyclic softening is confirmed to occur at and higher than εt = 0.30% following initial cyclic hardening to σmax = 330 MPa. At εt = 0.30%, the cyclic hardening starts earlier and the stress decrease after showing a peak of σmax is more remarkable than those at εt = 0.40 and 0.50%. It is also noted that cyclic hardening appears again after the cyclic softening. Such softening was also observed to occur in Ni3Fe single crystals [24,25], though the stress decrease is more remarkable than that in Fe3Al. The accumulated plastic strain per cycle at εt = 0.30% is plotted

![Fig. 1. Schematic illustrations of magnetic measurement. (a) A circular disc for magnetic measurement. (b) High-field susceptibility in the approach to magnetic saturation.](image1)

![Fig. 2. Cyclic hardening curves of Fe–28.0 at% Al single crystals cyclically deformed at different εt. The arrows indicate the onset of cyclic softening.](image2)
against number of cycles in Fig. 3. At the initial stage, the plastic strain diminishes steeply as \( N \) increases. This is reasonable because cyclic hardening results in a decrease in plastic strain in total-strain-controlled tests [13]. After touching bottom, however, the plastic strain slightly increases followed by a second decrease. The increase in plastic strain corresponds to cyclic softening as shown in Fig. 2.

### 3.2. Deformation microstructure

Fig. 4 shows slip markings on the surface of Fe–28.0 at% Al single crystals cyclically deformed at \( \varepsilon_t = 0.50\% \). The primary slip system was confirmed to be \((\overline{1}01)\{111\}\). At \( N = 10 \) cycles, faint contrast of \((\overline{1}01)\) slip traces can be seen in Fig. 4(a); this means the crystal was homogeneously deformed. On the other hand, coarse slip bands are formed at \( N = 10^2 \) cycles (Fig. 4(b)); strain localization occurs in the slip bands which may act as a nucleation site for a fatigue crack in Fe\textsubscript{3}Al. In addition, cross-slip to the other plane seems to occur frequently in the slip bands. The formation of slip bands is believed to be closely related to cyclic softening in Fe\textsubscript{3}Al; the bands become coarser with increasing \( N \) as shown in Fig. 4(c) and (d). At and higher than \( N = 10^3 \) cycles, a secondary slip of \((101)\{111\}\) also appears. Activation of the secondary slip system and the interaction between the primary and secondary slips may result in the cyclic hardening at the last stage of fatigue.

Fig. 5 shows deformation substructures parallel to the primary \((\overline{1}01)\) slip plane in Fe\textsubscript{3}Al single crystals cyclically deformed at \( \varepsilon_t = 0.50\% \). Screw dislocations of \( b = \frac{1}{2}\{111\} \) are dominantly observed at both \( N = 10^2 \) and \( 10^3 \) cycles as shown in Fig. 5(a) and (b). In a weak-beam image at high magnification (Fig. 5(c)), there is a pair of superpartials with \( b = \frac{1}{4}\{111\} \). This suggests that the superpartial pair moves dragging NNN APB, though four-fold dissociation of \( b = \frac{1}{2}\{111\} \) is generally observed to occur in Fe\textsubscript{3}Al [10,12]. The deformation substructure nearly perpendicular to the primary \((\overline{1}01)\) slip plane is also shown in Fig. 6. At \( N = 10^2 \) cycles, dislocation arrangement is very planar as shown in Fig. 6(a), while in Fig. 6(b) it is found to occur at \( N = 10^3 \) cycles resulting in the formation of dislocation bands. The formation of dislocation bands generally requires a frequent cross-slip of screw dislocations and is believed to be responsible for the cyclic softening at the middle stage of fatigue in Fe\textsubscript{3}Al single crystals.

![Fig. 3. Change in plastic strain with number of cycles in Fe–28.0 at% Al single crystals cyclically deformed at \( \varepsilon_t = 0.30\% \). The arrow indicates the onset of cyclic softening.](image1)

![Fig. 4. Optical micrographs of Fe–28.0 at% Al single crystals cyclically deformed at \( \varepsilon_t = 0.50\% \); (a) \( N = 10^1 \), (b) \( N = 10^2 \), (c) \( N = 10^3 \) and (d) \( N = 9049 \).](image2)

![Fig. 5. Dislocation structure parallel to primary \((\overline{1}01)\) slip plane in Fe–28.0 at% Al single crystals cyclically deformed at \( \varepsilon_t = 0.50\% \). (a) \( N = 10^2 \) cycles, \( g = 202 \) (b) \( N = 10^3 \) cycles, \( g = 202 \) and (c) weak beam image and the schematic illustration, \( g = 202, g/3g \) condition.](image3)
3.3. Magnetic observation of dislocation substructure

3.3.1. Theoretical approach to magnetic anisotropy induced by deformation

In ferromagnetic intermetallic compounds, two types of magnetic anisotropy are induced by deformation [14–21]. One is due to atomic rearrangement near APB [14–16], the other is due to internal strain around dislocations [17–21]. Fig. 7 shows schematic illustrations of the atom arrangement on (101) slip plane before and after deformation. Before deformation, there is neither a first-nearest nor a second-nearest Al–Al bond as shown in Fig. 7(a). Two types of APB, so called NN- and NNN-APBs are created after deformation, as shown in Fig. 7(b) and (c), respectively. In contrast to the perfect D0₃ crystals, first-nearest and second-nearest Al–Al bonds appear in NN- and NNN-APBs, respectively. It should be emphasized that Fe–Fe and Al–Al bonds are aligned parallel to [101] direction in NNN-APB shown in Fig. 7(c). Alignment of these bonds results in strong magnetic anisotropy of which the easy and hard directions are [101] and [010], respectively. The coefficient of magnetic anisotropy energy ($K_{\text{APB}}$) is written as follows [15,16]

$$K_{\text{APB}} = \frac{l_0 S r_0 \rho}{2\sqrt{2} a_0} F_{\text{APB}},$$

where $l_0$ is the dipole–dipole interaction, $S$ is the degree of long-range order, $r_0$ is the average width of APB, $a_0$ is the lattice constant, and $\rho$ is the dislocation density. $F_{\text{APB}}$ is the anisotropy function depending on the direction of magnetic field. It is also well known that an elastic strain field develops around dislocations depending on the type. Interaction between the internal strain and magnetic moment through the magnetoelastic coupling energy results in the rotation of magnetic moments around dislocations, so that the interaction induces a magnetic anisotropy. Brown, Seeger and Kronmüller [17–21] investigated the magnetic anisotropy based on micromagnetics. From their calculation, high-field susceptibility ($\chi_E$) in the approach to saturation is given by Refs. [17–21]

$$\chi_E = \frac{G^2 b^3 \rho}{64 \pi^2 H^3 I_s (1 - \nu)^2} \left( \ln\frac{R_0}{2 l_H} - 0.1728 \right) F_{\text{disl}},$$

where $G$ is the shear modulus, $b$ the length of Burgers vector, $I_s$ the saturation magnetization, $H$ the external magnetic field, $\nu$ the Poisson’s ratio, $R_0$ the average distance between dislocations, $l_H$ the exchange length and $F_{\text{disl}}$ the anisotropy function due to dislocations. $F_{\text{disl}}$ depends on the type of dislocations such as edge or screw. The anisotropy functions of $F_{\text{APB}}$ and $F_{\text{disl}}$ for several types of primary dislocation are plotted against the angle $\phi$ between primary slip direction of [111] and the direction of magnetic field in Fig. 8. From the theoretical calculation, the anisotropy functions of $F_{\text{APB}}$ and $F_{\text{disl}}$ can be represented by...
the following equation [23]

\[ F(\phi) = A \cos 2\phi + B \sin 2\phi + C \cos 4\phi + D \sin 4\phi + E, \]

(3)

where \( A, B, C, D \) and \( E \) are anisotropy coefficients depending on the type of defects as listed in Table 1. Since measured anisotropy is the overlap of these anisotropy functions, we can evaluate the type and density of lattice defects by analysing the anisotropy. The details are well described in our previous paper [23].

3.3.2. Analysis of measured anisotropy

Fig. 9 shows variation in high-field susceptibility with \( \phi \) in Fe3Al single crystals cyclically deformed at \( \varepsilon_0 = 0.50\% \). A strong anisotropy is obviously induced by fatigue in the crystals. At any cycle, the magnetic anisotropy exhibits two minima at around \( \phi = 60^\circ \) and \( 155^\circ \). The minimum at around \( 60^\circ \) is consistent with the magnetic anisotropy due to screw dislocations in Fe3Al. On the other hand, the magnetic anisotropy whose minimum is located at \( \phi = 155^\circ \) is due to atomic rearrangement near NNN-APB. Thus, the magnetic anisotropy is split into two components by the theoretical calculation. Since the coefficients \( C \) and \( D \) for NNN-APB are zero, the coefficients are only dependent on dislocations. In this case, we can determine the type and density of dislocations by solving simultaneous equations focusing on \( \cos 4\phi \) and \( \sin 4\phi \) terms. After that, APB-dependent anisotropy is obtained by excluding that due to dislocations from the measured anisotropy. Fig. 10 shows APB- and dislocation-dependent anisotropies extracted from the measured anisotropy in Fe3Al single crystals cyclically deformed at \( \varepsilon_0 = 0.50\% \). Two types of anisotropy were successfully separated from each other and the anisotropy due to edge dislocations could be neglected. As shown in Fig. 10(a), the amplitude of magnetic anisotropy due to NNN-APB increases rapidly with increasing cycles from \( N = 10^2 \) to \( 10^3 \) cycles; the anisotropy at \( N = 10^3 \) cycles is approximately six times larger than that at \( N = 10^2 \) cycles. In contrast, the dislocation-dependent anisotropy is almost constant regardless of \( N \) (Fig. 10(b)). From the magnetic anisotropy due to the screw dislocations, the density of screw dislocations could be evaluated by Eq. (2) as shown in Fig. 11. The density of the screw dislocations at \( N = 10 \) cycles is about \( 10^{13} \text{m}^{-2} \) and does not change with \( N \). This is because the plastic strain per cycle decreased rapidly with increasing \( N \) in the total-strain-controlled fatigue tests as shown in Fig. 2. The slight decrease in the density between \( N = 10^2 \) and \( 10^3 \) cycles may come from the dislocation rearrangement shown in Fig. 6(b). In any event, the calculated density is in fairly good agreement with TEM observation shown in Fig. 5. However, two types of magnetic anisotropy in cyclically deformed Fe3Al single crystals are seemingly inconsistent with each other. According to Eqs. (1) and (2), the amplitudes of both types of anisotropy are proportional to dislocation density, \( \rho \). However, magnetic anisotropy due to NNN-APB demonstrated a huge increase from \( N = 10^2 \) to \( 10^3 \) cycles, while that due to screw dislocations remained constant irrespective of \( N \). In our previous paper, we examined the magnetic anisotropy in cyclically deformed Ni3Fe single crystals. In the crystals, magnetic anisotropy due to APB was dominant at the initial stage of fatigue, while dislocation-dependent anisotropy became remarkable at the late stage. The change in magnetic anisotropy suggested that the degree of long-range order in Ni3Fe decreased during cyclic deformation. In contrast to Ni3Fe, magnetic anisotropy due to NNN-APB abruptly increased during fatigue, though that due to the screw dislocations was almost unchanged. The enhancement of magnetic anisotropy means the increase in the width of APB, \( r_0 \) in Eq. (1). The \( r_0 \) increases with disordering, while the decrease in the degree of order, \( S \) compensates for the variation of \( r_0 \).

| Types of defects         | Coefficients of anisotropy function |
|--------------------------|------------------------------------|
|                          | \( A \)    | \( B \)   | \( C \)   | \( D \)   | \( E \)   |
| NNN-APB                  | 0.1667    | -0.4714  | 0         | 0         | 0.5000    |
| Screw dislocation \((10^{-7})\) | 168.4    | -214.2   | 207.5     | 106.3     | 505.6     |
| Edge dislocation \((10^{-7})\) | 171.6    | -218.6   | -1.634    | -6.593    | 293.4     |

Fig. 8. Anisotropy functions for NNN-APB, screw and edge dislocations with \( b = [111] \) in Fe3Al with angle \( \phi \).

Fig. 9. Measured magnetic anisotropy in high-field susceptibility in Fe–28.0 at% Al single crystals cyclically deformed to different cycles at \( \varepsilon_0 = 0.50\% \).
In this case, the amplitude of magnetic anisotropy due to APB never increases by disordering. This suggests that disordering during fatigue is not likely to occur in Fe₃Al single crystals. According to Leamy et al. [26,27], the dragging of NNN-APB takes place even if the degree of D₀₃ order does not vary. If the shear stress of Fe₃Al exceeds the critical value \( \tau_c \), the pair of superpartials coupled by NN-APB can move, dragging NNN-APB

\[
\tau_c = \tau_0 + \frac{S^2 \gamma_{NNN}}{2b},
\]

where \( \tau_0 \) is the frictional stress of the superpartials, \( S \) is the degree of D₀₃ order, \( \gamma_{NNN} \) is the energy of NNN-APB and \( b \) is the magnitude of superpartial with \( b = 1/4[111] \). In this case, the APB area increases with both the dislocation density and the degree of order constant. Substituting each parameter to Eq. (4) (\( \tau_0 = 73 \) MPa, \( S = 0.8, \gamma_{NNN} = 80 \text{ mJm}^{-2}, b = 0.251 \text{ nm} \)), \( \tau_c \) was calculated to be 175 MPa. If the hypothesis is true, cyclic softening occurs at around \( \sigma_{max} = 350 \) MPa since the Schmid factor for primary (T01)[111] slip is 0.5. The cyclic softening at \( \epsilon_1 = 0.30\% \) began to occur above \( \sigma_{max} = 330 \) MPa as shown in Fig. 1. On the contrary, \( \sigma_{max} \) did not exceed 300 MPa at and below \( \epsilon_1 = 0.25\% \) resulting in the stress saturation after initial hardening. Therefore, the calculation is fairly consistent with the experimental data. Thus, the pair of superpartials moves dragging NNN-APB in the cyclic softening stage without disordering in Fe₃Al. The cross-slip of the superpartials becomes easy by the change in dislocation configuration, resulting in the dislocation rearrangement and the cyclic softening. The cyclic softening can appear when it overcomes the cyclic hardening. The cyclic hardening becomes strong as \( \epsilon_1 \) rises as shown in Fig. 2. Therefore, a marked decrease in maximum stress began early at \( \epsilon_1 = 0.30\% \), compared with that at \( \epsilon_1 = 0.40 \) and 0.50\%, as shown in Fig. 2.

4. Conclusions

Fatigue behaviour of Fe–28.0 at% Al single crystals was examined by means of TEM and the magnetic technique, and the following conclusions were reached

(1) In the total-strain-controlled fatigue tests, a weak cyclic softening was observed to occur in Fe–28.0 at% Al single crystals following initial cyclic hardening.

(2) The formation of dislocation bands was observed at \( 10^2 \) cycles, though the dislocation arrangement was very planar at the initial stage of fatigue. A pair of superpartials connected by NN-APB moved, dragging NNN-APB. The change in dislocation configuration may ease the cross-slip of dislocations resulting in the formation of dislocation bands. This also led to the cyclic softening in Fe₃Al single crystals.

(3) In Fe₃Al, magnetic anisotropy induced by cyclic deformation was composed of that due to atomic rearrangement near NNN-APB and internal strain around the screw dislocations. From the analysis of the measured anisotropy, the amplitude of APB-dependent anisotropy increased rapidly with increasing \( N \) from \( 10^2 \) to \( 10^3 \) cycles, while that due to screw dislocations remained almost constant during fatigue. This suggested that little disordering of D₀₃...
phase occurred during fatigue. The maximum stress of cyclically deformed Fe₃Al single crystals exceeded that required for superpartial pairs to drag NNN-APB resulting in the cyclic softening.

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