Serration of Grain Boundary in Ni–30Fe Alloy through High Temperature Deformation

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Grain boundary serration that evolved during high temperature deformation was investigated under various deformation conditions in a Ni–30Fe alloy. The maximum amplitudes of the serration at compressive strains of 0.13 and 0.34 were about 7 μm and 9 μm , respectively, at 1123 K. The amplitude decreased at lower deformation temperature and no serration was observed at 823 K. The amplitude of the serration increased linearly with an increase of strain in the first stage and became saturated above the strain of 0.2. TEM analysis showed that the grain boundary serration was closely related to the evolution of the sub-boundaries. The correlation between the development of the grain boundary serration and the size of the recovered equiaxed grain is discussed.

KEY WORDS: Ni–30Fe alloy; grain boundary serration; hot deformation.

1. Introduction

Many studies have shown that serrations develop at the grain boundaries during hot deformation for various materials like Al, Al alloys,^{1–3)} Ni alloys,⁴⁾ and stainless steels.⁴⁾

Serrated boundary plays an important role in the microstructural evolution during the hot deformation. The bulged area that is surrounded by the serrated boundaries can act as a nucleus for recrystallization.⁵⁾ The serration is believed to be responsible for the formation of multi-variant ferrite grains that are nucleated and transformed along the austenite grain boundary.⁶⁾ Furthermore, the serration prevents grain boundary sliding, resulting in the enhancement of the creep resistance and the fatigue resistance.⁴⁾

The ledge formation by the transgranular slip^{7,8)} or the grain boundary migration that interacted with the sub-boundary formation⁹⁾ has been thought to cause the serrated boundary. Recently, Hasegawa *et al.*⁹⁾ showed that the interaction between the grain boundary and the sub-boundary is mainly responsible for the development of the grain boundary serration during high temperature deformation for Al alloys by direct observations with TEM. Since the recovery process occurs preferentially for materials such as Al with high stacking fault energy (SFE), the formation of the dislocation cell structure and the sub-boundary and their interaction with the grain boundary seem to be plausible.

However, it is not clear whether the same mechanism occurs for materials with low SFE such as austenitic iron where recrystallization is the preferential restoration process. The present authors have investigated the so-called geometric recrystallization during the severe deformation at rela-

tively low temperatures for the Ni–30Fe alloy and reported that the interpenetration of the serrated boundary results in a recovered equiaxed grain (REG) structure without discontinuous recrystallization.¹⁰⁾ In this phenomenon, the grain boundary serration has a great influence on the size of REG.

The present paper primarily aims at investigating the mechanism of the grain boundary serration for the Ni–30Fe alloy with the SFE of 75 mJ/m².¹¹⁾ The SFE is similar to that of the austenite for low carbon steels and is about a half of that for Al, 166 mJ/m².¹²⁾ In addition, the present paper clarifies the role of the grain boundary serration as the controlling factor for the size of REG. The development of the grain boundary serration has been investigated by varying the deformation temperature and the applied strain for these purposes.

2. Experimental

Table 1 shows the chemical compositions of the test material. **Figure 1** shows the TMP (thermo-mechanical processing) paths for the hot compression tests. The hot compression tests were conducted on 12 mm (T)×15 mm (W)×20 mm (L) specimens with a hot deformation simulator.¹³⁾ The specimen was heated to each deformation temperature at a heating rate of 5 K/s, held for 5 s, and then deformed

Table 1. Chemical compositions of test alloy (in wt%).

C	Mn	Si	P	S	Ni	Fe
0.002	<0.002	0.008	0.002	0.001	69.8	Bal.

Fig. 1. TMP paths for hot compression tests.

Fig. 2. Calculated compressive strain distributions and measured one at a deformation temperature of 1123 K with a strain rate of 0.1/s.

with various reduction ratios at a nominal strain rate of 0.1/s. To freeze the deformed structure, the specimen was quenched to room temperature by a water jet just after the deformation. Metallographic analysis was performed with an optical microscope and a transmission electron microscope (TEM). Microstructural observation was carried out on sections cut through the center of the specimen and parallel to the compression axis as shown in Fig. 1.

The distributions of the compressive strain in specimens deformed with various reduction ratios were evaluated by finite element method (FEM). Figure 2 shows the distribution of the compressive strain calculated by FEM when the initial thickness of the specimen was 12 mm. As shown in

Fig. 2, the measured compressive strain with the screw embedded specimen¹⁴⁾ showed a good agreement with the calculated strain.

2.1. TMP Path 1

The development of the serration owing to strain was investigated in this path. The deformation temperature was 1123 K. From Fig. 2, the nominal reduction ratios were 4.5%, 8.8%, 13% and 21%, which corresponded to the compressive strains of 0.07, 0.13, 0.19 and 0.34, respectively, at the center of the specimen.

To study the characterization of the grain boundary, the development of other deformation structures like microbands or shear bands should be avoided in TEM observation of the grain boundary serration and the dislocation structures in the deformed specimens. Since the band structures are not common when the strain is less than 0.1 but prominent when the strain is above 0.2,¹²⁾ the compressive strain in the specimen must be less than 0.2. From Fig. 2, the nominal reduction ratio of 8.8% was adopted for the TEM characterization of the microstructures with the serrated boundaries, so that the compressive strain at the specimen center is between 0.1 and 0.2.

2.2. TMP Path 2

The development of the serration owing to the deformation temperature was investigated in this route. The deformation temperature was varied from 823 K to 1123 K. The nominal reduction ratios of 8.8% and 21% were adopted, corresponding to the compressive strains of 0.13 and 0.34, respectively.

3. Results

3.1. Effect of the Compressive Strain on the Serration Amplitude

Figure 3 shows the optical micrographs of the specimens deformed at 1123 K with the compressive strains of 0.07, 0.13, 0.19 and 0.34. Compared with the microstructure of the un-deformed specimen, the grain boundary serration develops clearly even at a compressive strain of 0.07. This means that the serration starts to evolve in a fairly early state of deformation. Figure 4 shows the meaning of the amplitude of the grain boundary serration and the change of the maximum amplitude by the compressive strain. The amplitudes of the serration are measured from the optical micrographs of each specimen. The maximum amplitude means the observable maximum amplitude of the serration in the analyzed optical micrographs. The conception of the maximum amplitude of the serration rather than that of the average value of the amplitude is adopted in present study because of its clearness. The frequency of the serration is not considered in present study. The maximum amplitude of the serration is about 4 μm at the compressive strain of 0.07 and the amplitude monotonously increases to 8.5 μm until the compressive strain is 0.2. However, the serration amplitude seems to be saturated above the compressive strain of 0.2, while the initial grains are still compressed.

Fig. 3. Optical microstructures of specimens at a deformation temperature of 1123 K. (a) undeformed (b) compressive strain of 0.07 (c) compressive strain of 0.13 (d) compressive strain of 0.19 (e) compressive strain of 0.34

Fig. 4. The change of maximum amplitude of the grain boundary serration with various amounts of strain at 1123 K.

3.2. Effect of the Deformation Temperature on the Serration Amplitude

Figure 5 shows the optical micrographs of the specimens

deformed with the compressive strain of 0.13 through TMP path 2. Well-developed serrations with the amplitudes of a few microns are observed at the deformation temperature of 1123 K. When the deformation temperature decreases, the serration amplitude also decreases till the serrated boundaries are no longer observable at the deformation temperature of 823 K. Similar temperature dependence is observed with larger serration amplitude at a compressive strain of 0.34.

Figure 6 shows the change of the maximum amplitude of the serration according to the deformation temperature. For the compressive strains are 0.13 and 0.34, the amplitudes of the serration increase with increasing the compressive strain at any deformation temperature. If the results in Fig. 4 that the serration amplitude tends to be saturated above the compressive strain of 0.2 are considered, the amplitude for the compressive strain of 0.34 in Fig. 6 can be the saturated amplitude at each deformation temperatures in the present study. At 823 K the amplitude is almost zero for both compressive strains. The dependence of the serration amplitude on the deformation temperature implies that thermal activated process governs the grain boundary serration

Fig. 5. Optical microstructures of specimens deformed at a compressive strain of 0.13.
(a) 1123 K (b) 1023 K (c) 923 K (d) 823 K

Fig. 6. The change of maximum amplitude of grain boundary serration with various deformation temperatures.

during the hot deformation.

3.3. TEM Microstructures

Optical micrographs in Figs. 3 and 5 reveal the development of the serrations at the grain boundaries of the initial grains. The initial grains have sometimes twins. However, the twin boundaries look fairly straight even at high temperatures and with large strains. The microstructural characteristics around the serrated grain boundary and the twin boundary, which are observed by TEM, can be summarized as follows.

(a) Grain Boundary

A TEM micrograph showing the microstructural details around the grain boundary serration is represented in Fig.

7. The specimen was deformed at 1123 K with the compressive strain of 0.13. In this micrograph, the grain boundary serrations with the amplitude of about $0.5 \mu\text{m}$ and $1 \mu\text{m}$, respectively, are observed as indicated in a schematic drawing of Fig. 7. Although the observed maximum amplitude is about $7 \mu\text{m}$ under this deformation condition by optical microscopy, the TEM observation is restricted to a very small area, so the grain boundary serrations with rather small amplitudes can be observed. In Fig. 7, the substructures are seen inside the deformed grains, and the boundaries of the substructures contact with the tops or the bottoms of the serrations.

Selected area diffraction patterns (SADP) in Fig. 7 show that Region I has a different crystallographic orientation from those of Regions II, III and IV; however, Regions II, III and IV have almost the same orientation. This means that the boundaries between Regions II and III, and Regions III and IV, are sub-boundaries.

Thus the grain boundary serration is closely related with the sub-boundaries inside the deformed grain. In other words, the so-called grain boundary migration occurs by the interaction with the sub-boundaries.

(b) Twin Boundary

TEM micrographs in Fig. 8 show the twin boundaries in the same specimen observed in Fig. 7. It is interesting that the twin boundary does not have the serration like the grain boundary, but has the ledges with a height of less than 50 nm. Since the twin boundary of FCC materials, the $\Sigma 3$ boundary, has very low mobility, it may be difficult for the twin boundary to migrate by the interaction with the substructures in the deformed grain.

Fig. 7. TEM micrograph of grain boundary serrations and neighboring deformed substructures.

Fig. 8. TEM micrographs of ledges at the twin boundary and neighboring deformed substructures.

4. Discussion

The results of the present study can be summarized as follows:

- (i) The grain boundary serration is a temperature-dependent phenomenon.
- (ii) The serration amplitude becomes saturated above a certain compressive strain.
- (iii) The grain boundary serration develops in a close relation with the sub-boundary.

4.1. Activation Energy for Grain Boundary Migration

In the evolution of the grain boundary serration, the grain boundary migration that interacts with the sub-boundary plays a more important role than the ledge formation by the transgranular slip, since the former is due to a temperature-dependent process and the latter is not necessarily so.

If the ledge formation by the transgranular slip plays a major role in developing the grain boundary serration, the amplitude must be increased monotonously with an increase of the strain. This is not true in the present study.

Fig. 9. An analysis on activation energy for grain boundary migration.

The serration amplitude becomes saturated with a given compressive strain over a critical value.

The migration rate of the grain boundary (V) is related to the activation energy (Q) as follows.

$$V = V_0 \cdot \exp\left(-\frac{Q}{RT}\right) \dots\dots\dots (1)$$

Here, the activation energy for the migration corresponds to that for the self-diffusion. The migration rate at each deformation temperature can be calculated from the amplitude of the serration at given compressive strain and the time needed for the deformation. Since the maximum amplitude of the serration increases linearly until the compressive strain reaches to 0.2 as shown in Fig. 4, the serration amplitudes for the compressive strain of 0.13 are adopted in present study for the evaluation of the average migration rate of the grain boundary. For example, at the deformation temperature of 1123 K, the observed maximum amplitude of the serration is about $7 \mu\text{m}$ at a compressive strain of 0.13. The time needed for the total reduction of 8.8%, which corresponds to the compressive strain of 0.13 at the specimen center, is about 0.9 s. The average migration rate of the boundary, therefore, becomes about $7.8 \mu\text{m/s}$ and its logarithm is about 2.05.

Figure 9 shows the logarithm of the average migration rate of the grain boundary as a function of ($1/T$) for the compressive strain of 0.13. In Fig. 9 the slope of the broken line indicates the activation energy of the grain boundary diffusion of Ni, 105 kJ/mol.¹⁵⁾ One can recognize the activation energy for the grain boundary migration comparable value with this value, and confirm again that the development of the grain boundary serration is a diffusion process.

4.2. Saturation of Serration

As the deformation proceeds, the rearrangement of the dislocation structure such as the formation of the dislocation cell and the sub-boundary, starts to occur to achieve a low energy state. Finally, the subgrains reach a steady-state size after a critical strain. The interaction between the grain boundary and the sub-boundary is believed to be responsible for the development of the serration. Therefore, the evo-

Fig. 10. Critical strain for full development of the subgrain structure.¹⁶⁾

Fig. 11. A sketch of a microstructural evolution map during hot deformation.

lution of the serration keeps pace with that of the sub-boundary. In other words, the serration amplitude becomes saturated when the subgrain structure reaches the steady-state size after a given strain. Such critical strain can be determined from the flow curves as schematically shown in Fig. 10.¹⁶⁾ For the Ni-30Fe alloy, the critical strain is about 0.2–0.3 from the report by Hurley *et al.*¹⁷⁾ This critical strain for the full development of the sub-boundary coincides with the strain over which the serration amplitude becomes saturated.

4.3. Characteristic Deformed Structure

The present authors would like to emphasize another important matter. That is, the grain boundary serration finally develops a characteristic deformed structure in the Ni-30Fe alloy. In the previous study,¹⁰⁾ the characteristic microstructure, namely, the recovered equiaxed grain (REG), appears when the deformation temperature is too low for the dynamic recrystallization (DRX) to occur but the compressive strain is high enough, as schematically shown in Fig. 11. The REG structure can be evolved by the interpenetration of the serrated boundary and so the critical compressive strain is needed for the serrated boundary to impinge on each other. The resultant microstructures of REG structure

Fig. 12. The amplitude of the grain boundary serration (A) in comparison with the grain sizes (B) of different kinds of equiaxed grain structures (DRX and REG).

looks like equiaxed grains, which are surrounded by the high angle boundary but still have the deformation structures like deformation bands or dislocation cells within themselves. The phenomenon is similar to the 'geometric recrystallization' which is reported for Al and Al alloys.¹²⁾ The importance of the grain boundary serration in the evolution of the REG structure lies in the fact that the serration amplitude is closely related with the size of the REG. This is because the interpenetration of the serrated boundary plays a major role in the evolution of the REG structure. Figure 12 shows the changes in the experimental DRX grain size,¹⁰⁾ REG size¹⁰⁾ and the serration amplitude obtained in the present study according to the deformation temperature. The DRX grain size predicted by a function of Zener-Hollomon parameter¹⁸⁾ is also given. In Fig. 12, one can notice a good agreement of the experimental DRX grain size with the predicted one as far as the DRX is concerned. On the other hand, when the REG structure develops instead of the DRX structure at lower deformation temperature, the size of REG is larger than the predicted DRX grain size. Besides, it can be also noticed that the amplitude of the grain boundary serration has a potent influence on the size of REG. These results indicate that the formation mechanism for the equiaxed grain structure shifts from DRX to REG at lower deformation temperature with large enough strain and that the size of REG has close relation with the amplitude of the grain boundary serration.

5. Conclusions

The grain boundary serration of the Ni-30Fe alloy that evolved during hot deformation is investigated and the following conclusions can be drawn.

(1) The evolution of the grain boundary serration depends on the deformation temperature. It means that the development of the grain boundary serration is a thermally activated process.

(2) At a deformation temperature of 1123 K, the maximum amplitude of the grain boundary serration increases linearly with the strain at the first stage and then becomes saturated after a critical strain.

(3) The grain boundary migration by the interaction with the sub-boundary is suggested to play a major role in developing the serrated boundary.

(4) The evolution of recovered equiaxed grain (REG) that is different from dynamic recrystallization occurs at a lower deformation temperature with large enough strain. The REG size can be well explained by the amplitude of the grain boundary serration.

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REFERENCES

- 1) M. R. Drury and F. J. Humphreys: *Acta Metall.*, **34** (1986), 2259.
- 2) E. V. Konopleva, H. J. McQueen and E. Evangelista: *Mater. Charact.*, **34** (1995), 251.
- 3) W. Blum, Q. Zhu, R. Merkel and H. J. McQueen: *Z. Metallkd.*, **87** (1996), 14.
- 4) H. J. McQueen, N. D. Ryan, E. V. Konopleva and X. Xia: *Can. Metall. Q.*, **34** (1995), 219.
- 5) F. J. Humphreys and M. Hatherly: *Recrystallization and Related Annealing Phenomena*, Pergamon, Oxford, (1995), 206.
- 6) S. Torizuka, O. Umezawa, K. Tsuzaki and K. Nagai: *Tetsu-to-Hagané*, **86** (2000), 23.
- 7) R. D. Gifkins: *Acta Metall.*, **4** (1956), 98.
- 8) M. Tanaka: *J. Mater. Sci. Lett.*, **14** (1995), 381.
- 9) T. Hasegawa, K. Kawaguchi, T. Murata and T. Yakou: *Mater. Trans. JIM*, **32** (1991), 244.
- 10) D. W. Suh, S. Torizuka, A. Ohmori, T. Inoue and K. Nagai: *ISIJ Int.*, **42** (2002), 432.
- 11) W. Charnock and J. Nutting: *Met. Sci. J.*, **1** (1967), 123.
- 12) F. J. Humphreys and M. Hatherly: *Recrystallization and Related Annealing Phenomena*, Pergamon, Oxford, (1995), 23.
- 13) T. Inoue, S. Torizuka, K. Nagai and T. Ohashi: *Symp. on Mathematical Modeling in Metal Processing and Manufacturing in COM2000*, ed. by P. Martin *et al.*, Metallurgical Society of CIM, Ottawa, (2000), (CD-ROM).
- 14) S. Genda, Y. Kogo, S. Torizuka and K. Nagai: *CAMP-ISIJ*, **12** (1999), 1361.
- 15) E. A. Brandes and G. B. Brook: *Smithells Metals Reference Book*, Butterworth-Heinemann Ltd., Oxford, (1992), 13.
- 16) F. J. Humphreys and M. Hatherly: *Recrystallization and Related Annealing Phenomena*, Pergamon, Oxford, (1995), 363.
- 17) P. J. Hurley, B. C. Muddle, P. D. Hodgson, C. H. J. Davies, B. P. Wynne, P. Cizek and M. R. Hickson: *Mater. Sci. Forum*, **284-286** (1998), 159.
- 18) I. Savatori, T. Inoue and K. Nagai: *ISIJ Int.*, **42** (2002), 744.