Dynamic Recrystallization Behavior in a Low-carbon Martensite Steel by Warm Compression

Jianhong Li,1) Pingguang Xu,1) Yo Tomota1) and Yoshitaka Adachi2)

1) Graduate School of Science and Engineering, Ibaraki University, Hitachi, Ibaraki 316-8511 Japan.
2) Innovative Materials Engineering Laboratory, National Institute for Materials Science, Tsukuba, Ibaraki 305-0047 Japan.

Received on February 19, 2008; accepted on April 24, 2008

The dynamic recrystallization behavior during warm compression for a low carbon martensite steel was investigated to make clear the effects of initial martensite block size, compression strain and pre-tempering before compression. It is found that the average size of recrystallized ferrite grains is influenced neither by the initial martensite block size (austenitizing temperature) nor by the amount of compression strain. The pre-tempering before compression shows two competitive effects: cementite particles precipitated during pre-tempering at 600°C promotes the occurrence of dynamic recrystallization while the decrease in dislocation density during pre-tempering at a higher temperature delays the dynamic recrystallization. Dispersed cementite particles suppress ferrite grain growth. Hence, there is an optimum tempering condition before warm-compression in order to obtain fine grained microstructure.

KEY WORDS: martensite; dynamic recrystallization; ultrafine grained steel; warm compression; pre-tempering.

1. Introduction

The dynamic recrystallization is one of the effective ways to refine microstructure of steel. Torizuka et al. have made a systematic study on warm compression or warm rolling for a conventional low-carbon ferrite–pearlite steel and reported fine grained ferrite–cementite microstructure is obtained through continuous dynamic recrystallization during severe deformation up to a true strain of 3–4.1,2) However, the severe deformation with a high Zener–Hollomon parameter \( Z \) is not easy to apply to the commercial production of steel plates or sheets. Here, \( Z \) is given by

\[
Z = \dot{\varepsilon} \cdot \exp \left( \frac{Q}{RT} \right)
\]

where \( \dot{\varepsilon} \), \( Q \), \( R \) and \( T \) refer to strain rate, activation energy, gas constant, and absolute temperature, respectively.

In 1972, Miller reported that ultrafine grained ferrite-austenite microstructure could be made by cold-rolling followed by annealing or warm working for Ni(–Mn)–C martensite steels, claiming that martensite is superior as the initial microstructure to refine ferrite grain size by thermomechanically controlled processing (TMCP).3) Many researchers have confirmed that the cold rolling and annealing or warm working of lath–martensite is very effective to make an ultrafine microstructure with grain size of less than 1 \( \mu \)m.4,7) Bao et al. have shown that the dynamic recrystallization occurs at a low critical strain by the warm compression of a low-carbon martensite steel.8) Furuhara et al. have found that a higher carbon martensite is more effective to reduce the critical strain for full dynamic recrystallization.9,10) Xu et al. reported that the precipitation of carbon-rich, i.e., hard austenite grains promoted the dynamic recrystallization during warm compression for a 17Ni–0.2C martensite steel.11)

In ferrite or ferrite–pearlite initial microstructure, it is well known that the average size of fully recrystallized grains is dependent on \( Z \) but independent of the initial grain size and the total strain.1,12,13) The average grain size of dynamic recrystallized microstructure is also confirmed to be dependent on \( Z \)-value in case of martensite initial microstructure.13) The other points are however not clear. Then, this paper is aiming at answering the following questions concerning so called continuous dynamic recrystallization in martensite steels.

1. Does the martensite block size influence the recrystallized ferrite grain size?
2. Does the amount of plastic strain influence the recrystallized ferrite grain size?
3. How does pre-tempering before warm compression influence the dynamic recrystallization behavior?

The reasons why martensite is excellent as the initial structure for dynamic recrystallization have been suggested to be high dislocation density, fine grain size and potency for precipitation during warm compression. Because pre-tempering changes such microstructural conditions, the above question (3) was focused in this study.

2. Experimental Procedures

A low-carbon steel (JIS SM490: 0.16C–1.43Mn–0.41Si–
0.014P–0.004S–0.01Cu–0.027Al–0.028N in mass%) was used. The transformation temperatures at a heating and cooling rate of 5°C/s were measured by dilatometry and the results were 737°C for Ac₁, 860°C for Ac₃ and 622°C for Ar₁, respectively. The 10/H₁₁₀₀₃₁₅ mm steel bars were austenitized at 900°C, 1000°C or 1150°C for 3.6 ks followed by water quenching to obtain full martensite microstructure. Cylindrical compressive specimens with 6 mm in length and 4 mm in diameter were prepared by spark cutting. The compression test was performed using a hot-compression rig with a heating system of direct electrical resistance and temperature was measured by R-thermocouples welded on the surface of a compression specimen. The specimens were heated up to 600°C or 650°C with a heating speed of 5°C/s, held there for 1 s, compressed by true strains (ε) of 0.3, 0.55 or 0.7 at 1.7 × 10⁻³ s⁻¹, and then quenched into water. Some specimens were pre-tempered at 600°C or 650°C for 3.6 ks or 18 ks before the warm compression. Figure 1 illustrates the procedures of heat-treatment and warm compression. The deformed specimens were sectioned along the compressive direction and the central area of the sectioned plane was observed with a field emission scanning electronic microscope (FE-SEM). After mechanical polishing, grain orientation characteristics were measured by electronic backscattering diffraction (EBSD) at a pitch of 200 nm for the initial martensite microstructures and 100 nm for the deformed microstructures. The average size of recrystallized ferrite grains surrounded only by high angle grain boundaries (hereafter called Type I grain according to Torizuka’s definition) was statistically analyzed by the ASTM linear intercept method. The mean block size of martensite and the average size of recrystallized ferrite grains surrounded partially by high angle grain boundaries (hereafter called Type II grain) were analyzed by the TSL orientation image mapping (OIM) software.

3. Results and Discussion

3.1. Dynamic Recrystallization Behavior in As-quenched Martensite Steel

Bao et al. have investigated the dynamic recrystallization behavior during warm compression of as-quenched martensite steel, SM490, at a compressive strain of 0.7 and found that the dynamic recrystallization occurs in specific conditions of strain rate and temperature. In order to investigate the effects of initial microstructure, compressive strain and pre-tempering, two compression temperatures, i.e. 600°C and 650°C at 1.7 × 10⁻³ s⁻¹ were selected in this study according to Bao et al.’s results. Figure 2 shows an example of crystallographic orientation characteristics for as-quenched martensite (austenitized at 1000°C) and ferrite microstructure observed before and after warm deformation of a compressive strain of 0.55. The as-quenched martensite blocks are being destroyed during warm compression while equiaxed ferrite grains coexist with some recovered substructures (see Figs. 2(c) and 2(d)). This is nearly consistent with Bao et al.’s results. Although the as-quenched martensite specimen was rapidly heated to 600°C and immediately (holding for one second) compressed, tempering could not be avoided. Hence, the tempering effect will be discussed in Sec. 3.4.

3.2. Effect of Compressive Strain on Dynamic Recrystallization

The specimens quenched from 1000°C were subjected to various compressive strains at 600°C and 650°C, respectively. Figure 3 shows the characteristics of grain boundary misorientation of the ferrite microstructures observed in the warm-compressed specimens. The volume fraction of equiaxed ferrite grain surrounded only by high angle boundaries, Type I grain, increases with increasing of compressive strain and such equiaxed ferrite grains show little change in the average size, suggesting the occurrence of continuous dynamic recrystallization. Figure 4 depicts the change in grain size distribution with compressive strain. The coexistence of Type I grains (marked by solid arrows) and Type II grains (marked by open arrows) can be found in Fig. 3(a) while Type II grains are coarser and in pancake shape. With increasing of compressive strain, the number of Type II grains decreases. Figures 3 and 4 reveal that the
critical strain for full recrystallization at 600°C is about 0.7.

Figure 3(a) also shows that the dynamic recrystallization in the martensite steel initiates at a small strain of 0.3, confirming that the critical strain for the onset of dynamic recrystallization in martensite steels is much lower than that in conventional ferrite–pearlite steels (about 1.2 according to Torizuka et al.\textsuperscript{2}).

Figure 5 shows the changes in the average size of Type I+II ferrite grains obtained from the specimens subjected to various compressive strains at 600°C and 650°C, respectively. With increasing compressive strain, the average size of all ferrite grains (Type I+II) calculated by the OIM software decreases and approaches to the average size of Type I grains. On the other hand, the average size of Type I grains shows little decrease with increasing of compressive strain. In addition, the average size of Type I grains at 650°C is larger than that at 600°C, showing the Z-value influences the average size.

3.3. Influence of Initial Microstructure on Dynamic Recrystallization

Figure 6 shows the effects of austenitizing temperature on the optical microstructure and the characteristics of martensite block orientation. With elevating the austenitizing temperature, the prior austenite grains, martensite packets and blocks become coarser. The orientation maps exhibit that the average aspect ratio of martensite blocks decrease with increasing of austenitizing temperature while the average length increases. Therefore, the effective grain size determined from high angle grain boundaries is the smallest for the 900°C austenitized specimen. Although other microstructural factors might be changed by the austenitizing temperature, only the block size is here focused.

Figure 7 exhibits true stress–strain curves obtained by warm compression at 600°C with 1.7×10^{-3} s^{-1} for the above three specimens. The flow stress reaches a maximum value after yielding and then gradually decreases, probably due to the occurrence of dynamic recrystallization. The flow stress of the specimen quenched from 1150°C is the largest and that of the specimen quenched from 900°C is the smallest. Vickers hardness measured at RT for specimens quenched from 1150°C, 1000°C and 900°C are HV499, HV483, HV465, respectively showing the same order of flow stress in Fig. 7.

Figure 8 shows the orientation maps for the grain orientation and boundary misorientation characteristics of the above three specimens after warm compression (600°C, 1.7×10^{-3} s^{-1}, ε=0.7). The equiaxed fine ferrite grains are
obtained in all of the specimens. The small regions with low confidence index points at ferrite grain boundaries are speculated to be related to cementite particles precipitated during warm compression. The size distributions for martensite blocks and those for all (Type I/H1100/II) ferrite grains are summarized in Fig. 9. It should be mentioned that the scale of the abscissa for martensite is different with that for ferrite. The difference in block size for the three specimens is clearly recognized in the left figures but that for ferrite is small (see the right figures). That is, the average grain size of ferrite is commonly about 2 μm or less. It suggests that the effect of block size, i.e., austenitizing temperature, is negligible.

3.4. Effects of Pre-tempering Time and Temperature on Dynamic Recrystallization

Figure 10 shows the effect of pre-tempering conditions on SEM microstructures, orientation maps and boundary misorientation characteristics of martensite microstructures quenched from 1000°C. It can be found that the cementite particles were being precipitated during pre-tempering and the cementite volume fraction of a specimen tempered at 650°C was higher than that at 600°C. Vickers hardness was decreased from HV483 for the as-quenched martensite to HV259 after 3.6 ks pre-tempering at 600°C and then HV198 after 18 ks pre-tempering at 650°C. The decrease in hardness is speculated to mostly due to the decrease in
dislocation density and also due to microstructural change during tempering.

**Figure 11** compares true stress–strain curves of four specimens obtained during warm-compression at 600°C, $1.7 \times 10^{-3}$ s$^{-1}$. The shape of curves is similar and the maximum flow stress becomes lower with pre-tempering. **Figure 12** shows the corresponding SEM microstructures, orientation maps and boundary misorientation characteristics obtained at $\varepsilon=0.7$. It is interesting that the grain refinement by warm-compression is remarkable in the specimen pre-tempered at 600°C (see Figs. 12(b), 12(f) and 12(j)).

The equiaxed ferrite grains in the pre-tempered specimen at 650°C after warm compression are coarser than those at 600°C. The over-tempering brings the coarsening of cementite particles which must reduce the grain boundary

**Fig. 9.** Block size distribution in specimens quenched from different austenitizing temperatures (a), (c), (d) and grain size distribution after compression at 600°C by $\varepsilon=0.7$ (b), (d), (f).

**Fig. 10.** SEM microstructures, orientation image maps and boundary misorientation characteristics of the as-quenched and pre-tempered martensite specimens.

**Fig. 11.** Flow stress–strain curves obtained at 600°C without and with pre-temperin treatment.
pinning effect, and decreases in dislocation density which must suppress the occurrence of dynamic recrystallization. Hence, some suitable tempering is preferable in order to obtain ultrafine grained microstructure by warm-deformation of a martensite steel. The rapid heating and immediate warm-working employed in our previous paper is not necessary and the slow heating may be allowable in the engineering application of this process.

The effects of block size, dislocation density and cementite particles on the critical strain for the onset of dynamic recrystallization and recrystallized ferrite grain size are summarized in Table 1.

### Table 1. Effect of initial microstructure on dynamic recrystallization in martensite steel.

| Effect                        | Critical strain | Recrystallized grain size |
|-------------------------------|-----------------|---------------------------|
| Decrease in block size        | Little influence| Little influence          |
| Decrease in dislocation density| Larger          | Little influence          |
| Cementite particles           | Smaller (*)     | Smaller                   |

(*) It seems to be dependent on the size and volume fraction of cementite.

The dynamic recrystallization behavior in a low-carbon martensite steel was investigated to make clear the effects of strain, initial martensite block size and pre-tempering. The main results obtained are as follows:

1. The average size of recrystallized ferrite grains is influenced neither by the initial martensite block size nor by the amount of compression strain.
2. The cementite particles finely precipitated by pre-tempering at 600°C seem to promote the onset of dynamic recrystallization, while a decrease in dislocation density by pre-tempering at a higher temperature hinders the dynamic recrystallization.
3. Dispersed cementite particles suppress ferrite grain growth. Hence, there is an optimum tempering condition before warm-compression in order to obtain fine grained microstructure.

### REFERENCES

1. A. Ohmori, S. Torizuka, K. Nagai, K. Yamada and Y. Kogo: *Tetsu-to-Hagané*, 88 (2002), 857.
2. S. Torizuka: *Bull. Iron Steel Inst. Jpn.*, 10 (2005), 188.
3. R. L. Miller: *Metall. Mater. Trans.*, 3 (1972), 905.
4. K. Ameyama, N. Matsumura and M. Tokizane: *J. Jpn. Soc. Heat Treat.*, 28 (1988), 233.
5. R. Ueji, N. Tsuji, Y. Minamino and Y. Koizumi: *Acta Mater.*, 50 (2002), 4177.
6. T. Hayashi, S. Torizuka, T. Mitsui, K. Tsuzuki and K. Nagai: *CAMP-ISIJ*, 11 (1998), 1031.
7. T. Hayashi and K. Nagai: *Trans. Jpn. Soc. Mech. Eng. A*, 68 (2002), 1553.
8. Y. Z. Bao, Y. Adachi, Y. Toomine, T. Suzuki, P. G. Xu and Y. Tomota: *Tetsu-to-Hagané*, 91 (2005), 602.
9. B. PoorGanji, G. Miyamoto, T. Furuhara and T. Maki: Proc. ISUGS, ISIJ, Tokyo, (2007), 81.
10. T. Furuhara, T. Yamaguchi, S. Furimoto and T. Maki: *Mater. Sci. Forum*, 539–543 (2007), 155.
11. P. G. Xu, J. H. Li, Y. Tomota and Y. Adachi: submitted to ISIJ Int.
12. T. Maki: *CAMP-ISIJ*, 19 (2006), 410.
13. A. Ohmori, S. Torizuka, K. Nagai, K. Yamada and Y. Kogo: *Mater. Trans.*, 45 (2004), 2224.
14. Y. Z. Bao, Y. Adachi, Y. Toomine, T. Suzuki, P. G. Xu and Y. Tomota: *Scr. Mater.*, 53 (2005), 1471.