Effect of Solution Carbon and Retained Austenite Films on the Development of Deformation Structures of Low-Carbon Lath Martensite

Shigekazu MORITO,^{1)*} Takuya OHBA,¹⁾ Ananda Kurmar DAS,¹⁾ Taisuke HAYASHI²⁾ and Mai YOSHIDA¹⁾

1) Department of Materials Science, Shimane University, 1060 Nishikawatsu-cho, Matsue, Shimane, 690-8504 Japan.

2) Department of Materials Analysis, Shimane University, 1060 Nishikawatsu-cho, Matsue, Shimane, 690-8504 Japan.

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The deformation structure of low-carbon lath martensitic steels was analysed by transmission electron microscopy with Kikuchi pattern analysis. Retained austenite films on the martensite lath boundaries are transformed into high-carbon martensite films by light deformation. These high-carbon martensite films protect the martensite lath boundaries from reconstruction. Specific deformation structures of low-carbon lath martensite, such as kinked laths, irregularly bent lamellas and lamellar dislocation cells, were thus formed. Tempered lath martensite structures without retained austenite films easily disappeared by light deformation. The relationship between mechanical properties, such as the work-hardening ratio, and the development of the deformation structure was also clarified.

KEY WORDS: lath martensite; cold rolling; deformation structure; transmission electron microscopy/Kikuchi pattern; crystallography, low-carbon low-alloy steels.

1. Introduction

Martensitic steels normally do not undergo deformation after quenching and tempering because of their hardness and brittleness. However, low-carbon martensitic steels can be deformed. This study aims to determine the factors that affect the degrees of deformation in such steels.

The low-carbon martensitic steels have high work-hardening rate and can be directly used after being formed for industrial products without heat treatment, such as tempering.¹⁾ Furthermore, deformed martensite has a good initial structure for high-strength high-toughness steels, *e.g.* recrystallized deformed martensitic steels exhibit a good strength–elongation balance.²⁾

The high work-hardening rate and good strength–elongation balance observed in low-carbon martensitic steels are caused by inhomogeneous deformation structures. In these steels, the deformation structures include lamellar dislocation cells (LDCs), irregularly bent laths (IBLs) and kinked laths (KLs) regions. The LDCs regions in the deformation structure increase with strain,³⁾ *i.e.* martensite laths deform. In contrast, in ultra-low-carbon martensitic steels, the lath boundaries in a given martensite block partially disappear during the early stages of deformation. For medium and large strains, deformation may occur in cell blocks with dislocation cells,^{4,5)} and finally, LDCs appear in the specimens. Their development is similar to that of ferrite,⁶⁾ their defor-

mation structures are more homogeneous than those observed in low-carbon steels and their work-hardening rate is lower than that in low-carbon steels.

We know that the development of deformation structures depends on the carbon content because of the following reasons: 1) solution carbon atoms segregate on the martensite laths, arresting the dislocation movement and maintaining the boundaries,⁷⁾ 2) fine carbides formed by auto-tempering act as a barrier and 3) retained austenite films located between adjacent laths that hold the lath boundaries.⁸⁾ However, the main factor controlling the development of deformation structures is still unknown.

No method has been reported to date to identify this factor. To analyse the process, various specimens were prepared at various tempering temperatures from as-quenched specimens to separate the microstructures, such as retained austenite films and carbides, and the deformed microstructures were observed and compared to clarify the effective factors.

2. Experimental Procedures

The chemical compositions of the steel used in this study are Fe–0.20C–<0.01Si–1.98Mn–<0.001P–<0.0007S–0.032Al–0.0026B in mass%. To obtain a fully lath martensite structure, the specimens were quenched in water after austenitization at 1 473 K for 600 s, tempered at 473–973 K for 600 s and cold rolled to 0%–80%.

The specimens were then studied by optical and transmission electron microscopy (TEM; JEOL JEM-2010 operated at 200 kV), the observational directions being transverse to the

* Corresponding author: E-mail: mosh@riko.shimane-u.ac.jp
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rolling direction and normal to the rolling plane of the specimens. Local crystal orientation and structures were analysed by convergent beam Kikuchi diffraction patterns (CBKP).⁹⁾ To observe the lath boundaries by TEM, the laths were observed in parallel along their habit planes. To check the existence of lath and/or dislocation boundaries, the diffraction conditions of the TEM images were always investigated.

The dislocation densities were estimated from x-ray diffraction profiles (Cu $K\alpha_1$, 40 kV, 20 mA, 0.01° steps) by Williamson–Hall analysis.¹⁰⁾ The full width at half maxima of the 011, 112, 022 and 222 peaks were used. The estimated dislocation density in the as-quenched martensite was $1.46 \times 10^{15} \text{ m}^{-2}$, which is similar to that determined by TEM, namely, $1.6 \times 10^{15} \text{ m}^{-2}$.¹¹⁾

The mechanical properties were determined by Micro Vickers hardness (9.8 N, 15 s) and tensile tests. The gauge length, width and thickness of the specimens for the tensile tests were 8.5, 2.5 and 0.5 mm, respectively. The initial strain rate was $2.3 \times 10^{-2} \text{ s}^{-1}$, and the strain during the tensile test was measured with a strain gauge of length and resistance 2 mm and 120 Ω , respectively.

3. Results

3.1. Initial Microstructures in As-quenched and Tempered Specimens

The optical micrograph of the as-quenched specimen (shown in Fig. 1) indicates block and packet structures in the prior austenite grains. The specimen displays the typical morphology of low-carbon lath martensite. The prior austenite grain size, packet size and block thickness are 350,

Fig. 1. Optical micrograph of an as-quenched specimen etched with 3% nital.

Fig. 3. Dislocation densities of specimens before cold rolling: ○ as-quenched and ● tempered specimens.

150 and 3.4 μm , respectively.

Figure 2 shows TEM micrographs of as-quenched and tempered specimens. The as-quenched sample [Fig. 2(a)] shows fine laths with dislocations; whereas in the case of the specimens tempered at 473 and 573 K (Figs. 2(b) and 2(c) respectively), fine carbides appear on the lath boundaries and in the laths without coarsening. The dislocation structures in the specimens are comparable with those in the as-quenched specimens. The specimens tempered at 573 K contain spheroidal carbides with a size of $\sim 50 \text{ nm}$, as shown

Fig. 2. TEM micrographs of as-quenched and as-tempered martensite specimens: (a) As-quenched, (b) 473 K-, (c) 573 K- and (d) 923 K-tempered specimens.

in Fig. 2(d). In these specimens, new dislocation cells appear and lath boundaries partially vanish, *i.e.* the dislocations are rearranged by recovering.

The dislocation densities in the as-quenched and tempered specimens in **Fig. 3** were determined by X-ray diffraction. At tempering temperatures above 673 K, the disloca-

Fig. 4. TEM micrographs of as-quenched and 473 K-tempered lath martensite: (a) bright-field image of an as-quenched specimen and (b) corresponding dark-field image,¹²⁾ (c) bright-field image of a 473 K-tempered specimen and (d) corresponding dark-field image. Each dark-field image was taken from the $\bar{1}1\bar{1}$ austenite diffraction spot.

Fig. 5. TEM micrographs after 10% cold rolling: (a) as-quenched, (b) 473 K-tempered, (c) 573 K-tempered and (d) 923 K-tempered specimens.

tion densities significantly decreased from 10^{15} to 10^{14} m^{-2} .

Retained austenite films on the lath boundaries are found both in the as-quenched specimens and specimens tempered at 473 K, as shown in Fig. 4. Dark-field images constructed by austenite diffractions [Figs. 4(b) and 4(d)] indicate that the laths are covered with the retained austenite films. The thickness of the films is 1–3 nm.

3.2. Development of the Deformation Structures

The microstructures of cold-rolled specimens were observed by TEM. Figure 5(a) shows that no deformed microstructures are found in the 10% cold-rolled microstructure of as-quenched specimens. The TEM micrograph obtained after 10% cold rolling of a specimen tempered at 473 K [Fig. 5(b)] also indicates non-deformed microstructures.

For the specimens tempered at 573 K, martensite laths exist in the deformed specimen, although the laths are divided by dislocation walls, as seen in Fig. 5(c). In the deformed 923 K-tempered specimens, martensite laths are subdivided and their boundaries partially vanish, as shown in Fig. 5(d). These results indicate that lath martensite in high-temperature tempered specimens is reconstructed into the dislocation cell structure even at 10% cold rolling.

Previous studies did not show retained austenite regions for all specimens. Figures 6(a) and 6(b), which represent a bright-field TEM image and the corresponding selected-area electron diffraction patterns in the 10% cold-rolled as-quenched specimen, indicate that the retained austenite film is not found after 10% cold rolling. However, there are film-like contrasts on the lath boundaries, which correspond to

Fig. 6. (a) TEM micrograph of a 10% cold-rolled as-quenched specimen and (b) related electron diffraction pattern with indexes. The white broken lines are former retained austenite films, and A–E indicate the points studied by TEM/KP.

Fig. 7. TEM micrographs after cold rolling: (a) 30% cold-rolled as-quenched, (b) 50% cold-rolled 473 K-tempered, (c) 30% cold-rolled 573 K-tempered and (d) 30% cold-rolled 923 K-tempered specimens.

retained austenite film regions, indicated as white broken lines in Fig. 6(a). CBKP measurements were performed to analyse the phase and crystal orientation in the films. For the analyses, each phase in regions A–E in Fig. 6(a) is indexed as a body-centred cubic phase, *i.e.* the film regions are ferrite or martensite. The misorientation angles between the adjacent regions, A–B, B–C, C–D and D–E, are 1.1° , 7.1° , 1.1° and 6.4° , respectively. The other observations also lead to the same results. The measurements indicate that the misorientation angles between one side of the films and the adjacent laths are less than 3° . These analyses indicate that the retained austenite films transform into ferrite or martensite films by light deformation.

The development of the deformation structure in each specimen is different. **Figure 7** shows TEM micrographs after 30%–50% cold rolling of the as-quenched and tempered specimens. The micrographs in the as-quenched and tempered specimens (473 K) [Figs. 7(a) and 7(b)] contain kinked and/or sheared lath regions.^{3–5} No lath boundaries are observed in the 30% cold-rolled 573 K-tempered specimens [Fig. 7(c)], except for some boundaries indicated by white broken lines. This means that the lath boundaries are

destroyed. In the 923 K-tempered specimens, the 30% cold-rolled specimens are fully occupied by deformed microstructures [Fig. 7(d)].

After 80% cold rolling, all specimens uniformly contain LDCs (**Fig. 8**). The thickness of lamellar boundaries in the specimens increases with tempering temperature. **Figure 9** shows the relationship between dislocation cell thicknesses, *i.e.* lath thickness and lamellar spacing, and tempering temperature. The lamella spacing suddenly increases when the specimens are tempered at 473 and 573 K, and differences in the lamellar spacing indicate differences in the development of the deformation structure, with or without reconstruction.

Figure 10 shows stress–strain curves around the yield stress. The largest yield stress is measured for the specimen tempered at 673 K, and the upper yield point is observed in the curve only. The yield stress decreases with increasing tempering temperature, except for the 673 K-tempered specimen. After yielding, the work-hardening rate decreases with increasing tempering temperature, except for the 473 K-tempered specimen, whose hardening rate is the same as that of the as-quenched specimen.

Fig. 8. TEM micrographs after 80% cold rolling: (a) as-quenched, (b) 473 K-tempered, (c) 573 K-tempered and (d) 923 K-tempered specimens.

4. Discussion

The nature of lath boundaries at various tempering temperatures can be described as follows: I) retained austenite films exist in the as-quenched specimens, II) retained austenite films and fine carbides exist in the 473 K-tempered speci-

Fig. 9. Relationship between lath thicknesses (○) and elongated dislocation cell thicknesses in 80% cold-rolled specimens (■).

Fig. 10. Stress–Strain curves around yield points.

mens, III) fine carbides exist in the 573 K-tempered specimens and IV) coarse carbides exist in the 923 K-tempered specimens. To classify the specimens according to the development of the deformation structure, KLs and IBLs are developed in the as-quenched and 473 K-tempered specimens (types I and II), whereas the lath martensite structure is reconstructed to the deformation structure in the specimens tempered at temperatures above 573 K (types III and IV). The differences in the deformation structures are caused by retained austenite films, *i.e.* the films maintain the lath boundaries, and the development of the deformation structure in type III specimens is slower than that in type IV. The reason is that there are fine carbides in type III specimens, which arrest the destruction of lath martensite, although the effect is not stronger than that of the retained austenite films.

The retained austenite films in the as-quenched and 473 K-tempered specimens are an effective factor for controlling the deformation structure, although the films transform by light deformation. The retained austenite films in the as-quenched specimens contain 1 mass% carbon atoms,¹²⁾ and they stabilize the austenite phase. Upon 10% cold rolling with a strain rate of $\sim 2 \text{ s}^{-1}$, which is higher than the carbon diffusion, the films transform into martensite films, with crystal orientations similar to the adjacent laths. This means that martensite films with 1 mass% carbon atoms maintain the martensite lath boundaries. During deformation, the dis-

location movement is arrested by the high-carbon martensite films because of their hardness, and KLs and IBLs are induced by inhomogeneous strain. If the dislocation movement is arrested strongly, cracks will be induced in the martensite films. However, high-carbon martensite films are not a strong barrier for the dislocation movement because the crystal orientation of the films is near to adjacent laths. Retained austenite films in ultra-low-carbon steels also transform into martensite films with crystal orientations similar to those of the adjacent laths, and the ultra-low-carbon martensite films are not a barrier to the dislocation movement.¹³⁾ Thus, high-carbon martensite films are the most important phase to arrest the destruction of lath martensite.

As shown in Fig. 9, the lamella spacing after 80% cold rolling significantly changes between 473 and 573 K. One of the reasons is the difference in the development of deformation structures. In the as-quenched and 473 K-tempered specimens, dislocation cells are developed by multiple slips caused by high-carbon martensite films. In the higher-temperature tempered specimens, the deformation structures are reconstructed from lath martensite, and their development is delayed. The dislocation densities also decrease with increasing tempering temperature. The increment in lamellar spacing from 573 K- to 923 K-tempered specimens is only $0.8 \mu\text{m}$, whereas the decrease in dislocation density is $\sim 10^{15} \text{ m}^{-2}$. It means that the latter parameter is not an effective barrier in the development of deformation struc-

Fig. 11. Schematic illustrations of the development of deformation structures in low-carbon lath martensite: (a) as-quenched, low-temperature and (b) high-temperature tempered specimens.

tures.

Figure 10 shows that the work-hardening rate decreases for the 573 K-tempered specimens, and the yield stress suddenly increases for the 673 K-tempered specimens. The reason for the former result is a change in the development of deformation structures, from a deformed lath structure to a vanished and reconstructed lath structure, whereas the latter result suggests that mobile dislocations decrease by rearrangement during tempering.¹⁴⁾ At tempering temperatures above 673 K, the yield stress decreases because of carbide coarsening and dislocation reduction.

The present results and discussion are summarized in **Fig. 11** with the schematic illustrations grouped by typical deformation structures. Figure 11(a) shows the development of the deformation structure of as-quenched and 473 K-tempered lath martensite in low-carbon steels containing retained austenite films. The retained austenite films with 1 mass% C¹²⁾ transform into high-carbon martensite films after light deformation, and martensite laths remain after 10% cold rolling because the high-carbon martensite films between adjacent laths act as a barrier to the disappearance of lath boundaries. At higher strains, the martensite laths change to KLs, IBLs and LDCs in 30%–50% cold rolling. After 80% cold rolling, the deformation structures develop in LDCs regions. In high-temperature tempered specimens without retained austenite films, only fine or coarse carbides remain in the lath structure. The restricting dislocation movements by the carbides between adjacent laths are weaker than that of retained austenite films, and the carbides are not completely covered by lath boundaries. Thus, the martensite laths disappear, and dislocation walls are induced across the laths. After the disappearance of laths, the deformation structure develops in the same way as that of equiaxed ferrite.^{4,5)}

5. Conclusions

The microstructural changes during cold rolling of lath martensite in low-carbon steels were clarified and compared with those observed in low-carbon martensitic steels with and without retained austenite films. The following results were obtained:

(1) In as-quenched and low-temperature tempered specimens with retained austenite films, the microstructures continuously change from martensite laths to KLs, IBLs and LDCs. In high-temperature tempered specimens without

retained austenite films, the martensite laths vanish, and dislocation cells are reconstructed.

(2) Low-temperature tempered specimens retain the martensite laths after deformation.

(3) Retained austenite films transform into high-carbon martensite films after light deformation. The crystal orientations of the martensite films are similar to those of the adjacent laths, with a difference less than 3°.

(4) The specific deformation structures and their development in low-carbon lath martensite steels depend on the retained austenite films and high-carbon lath martensite films, which act as a barrier to the disappearance of lath boundaries. The fine carbides also act as a barrier, but are not as effective.

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