Original Research

Twin structure of the lath martensite in low carbon steel

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Pan Zhang [a], Yulin Chen [a], Wenlong Xiao [a,*], Dehai Ping [b], Xinqing Zhao [a,*]

[a] School of Materials Science and Engineering, Beihang University, Beijing 100191, China
[b] School of Materials Science and Engineering, Dalian Jiaotong University, 116028 Dalian, China

ABSTRACT

It has been well accepted that the martensites in quenched carbon steels exhibit two typical morphologies which are closely dependent on the carbon content, i.e. lath martensite in low carbon steels and lenticular martensite in high carbon steels. Based on conventional belief, the lath martensites in low carbon steels are with high density dislocations as the substructure, in contrast to twin substructure in lenticular high carbon martensite. In the present work, an intensive transmission electron microscopy investigation was made to characterize the microstructures of the lath martensite in a low carbon steel of 0.2 wt%C. It was found that lots of lath martensites consist of twin as their substructure, rather than high density dislocations. In addition, nanoscale precipitates cohering with ferrite matrix were found at the twin interfaces. The orientation relationships between the precipitates and the ferrite matrix are in good agreement with that of primitive hexagonal ω phase in titanium alloys and other bcc metals or alloys.
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1. Introduction

It has long been recognized that rapid quenching of face-centered cubic (fcc) structured austenite in steels might result in the formation of body-centered cubic (bcc) or body-centered tetragonal structured martensite [1]. As one of the most important solid phase transformations in steels, the martensitic transformations have been consistently receiving much attention because of their technological importance and academic significance for the research and development of advanced steels [1–3]. In order to regulate the martensitic transformations and improve the mechanical properties of martensitic steels, numerous alloying elements such as C, N, Ni, Cr, Mn, etc. are usually incorporated in steels. Tremendous experimental investigations on martensitic transformation in steels have indicated that the microstructures and the mechanical behaviors of martensitic steels are closely related to the type and the content of alloying elements. Carbon, as the most important and effective alloying element in steels, could significantly influence the martensitic transformation behavior of carbon steels, including the volume fractions, the morphologies and the mechanical properties of martensites [1,4].

Although martensites can exhibit different morphologies in a large variety of alloying steels, i.e. lath, lenticular, butterfly, thin-plate, etc., martensites in Fe–C steels feature two typical shapes, lath and lenticular, depending on carbon content [5]. It was generally accepted that the lath martensites form in low carbon steels with 0~0.6 wt%C, whereas lenticular martensite becomes dominant morphology in high carbon steels with above 1.0 wt%C [6]. Nevertheless, there are some anomalous results reported on the morphologies of martensites in carbon steels. For example, some researchers observed lath martensites in quenched ultra-high carbon steel with 1.4 wt%C [7,8]. In addition, a trace of twin structure was observed in lath martensites in low carbon steel with 0.1 wt%C [9]. As a matter of fact, the mechanism for the dependence of martensite morphologies on carbon content is not clear, although this is very interesting and important for understanding the martensitic transformation in carbon steels.

Actually, the morphology, the crystallography and the microstructure of martensite in steels have been investigated for decades by numerous techniques such as optical microscopy, transmission electron microscopy (TEM) and scanning electron microscopy including electron backscattered diffraction [2, 10–13]. It was well acknowledged that the lath and lenticular (or plate) martensites in carbon steels possess different substructures inside the corresponding martensite variants, i.e. the lath martensites are with high density dislocations and lenticular martensites consist of numerous twins. In the early TEM observation on lath martensites of low carbon steels (< 0.5 wt%C), Kelly and Nutting claimed that the dislocations in lath martensites are with high density of the order of 10^{11}–10^{12} cm^{-2}, comparable to the dislocation density in very heavily cold-worked alloys [14,15]. Such

high density of dislocations makes it impossible to analyze clearly the planes on which the dislocations occur or determine the Burgers vectors of these dislocations [14,15]. However, except for the steels with ultralow carbon content [4], 0.0026% for example, no researchers observed distinguishable high density dislocations in the martensites of low carbon steels with the carbon content greater than 0.1 wt%C. Note that low carbon martensitic steels perform good toughness even at as-quenched state with full lath martensite structure(or containing very few residual austenite phase). Apparently, this result is not supportable to the proposition that the lath martensite is with high density of dislocations comparable to severely deformed microstructures. Although some dark contrast areas were frequently observed inside the lath martensites on TEM observations, no convincing evidence was achieved to demonstrate that these contrast areas arise from high density of dislocations. In the present study, we carried out an intensive TEM investigation on low carbon steel of 0.2 wt%C, in attempt to clarify the microstructures of the lath martensite. It was suggested that the lath martensites of the present study are composed of twins as their substructure, rather than high density dislocations. In addition, the twin structures of the lath martensites were probed, and nanoscale precipitates cohering with ferrite matrix at the twin boundaries were observed.

2. Material and methods

Commercial low carbon steel with composition of C~0.20 wt%, Mn~0.85 wt%, Cr~1.08 wt%, Fe~ balance was selected in the present study. According to a model for predicting the martensitic transformation starting temperature (M_s), $M_s(K) = 731 - 227(C+N) - 17.6Ni - 22.5Mn - 17.3Cr - 16.2Mo$, the M_s of the steel in the present study was calculated to be around 647 K [16]. The samples of $10 \times 10 \times 0.5$ (mm^3) were sealed in quartz tube under Ar atmosphere and austenitized at 1373 K for 30 min, followed by quenching into brine at 298 K. The specimens for TEM observation were prepared by grinding the discs to thickness of about 50 μm, and electro-polishing in a twin-jet electro-polisher with a chemical solution of 10% HClO$_4$ and 90% ethanol at 253 K. Phase constitutions were determined by X-ray diffraction with Cu Kα radiation at an accelerating voltage of 40 kV and a current of 200 mA. The microstructural observations were carried out on an optical microscope and a transmission electron microscope (JEM 2100F) operated at 200 kV.

3. Results and discussion

Because of the limited hardenability of low carbon steels, X-ray diffraction was conducted to inspect the constituents of the quenched samples. In order to analyze the diffraction of the quenched sample with 0.2%C with that of pure iron without carbon, Fig. 1 gives the diffraction profiles of these samples with same treatments, i.e. austenization at 1373 K for 30 min, followed by quenching into brine at 298 K. One can see from the Fig. 1 that no trace of austenite or carbide was detected in the diffraction patterns, indicating that full martensitic microstructure forms in the 0.2%C sample during the quenching.

Fig. 2 shows the optical microscopy image and bright-field TEM micrograph of the quenched low carbon steel sample, in which typical morphology of lath martensite are observable, with the crystallographic features of packet and blocks. Fig. 2(b) indicates that under certain direction of incident electron beam, parallel martensite variants exhibit different contrast, i.e. bright blocks and dark blocks. Especially, in the dark martensite blocks, one can see large amounts of dotted black structures. Note that these dotted
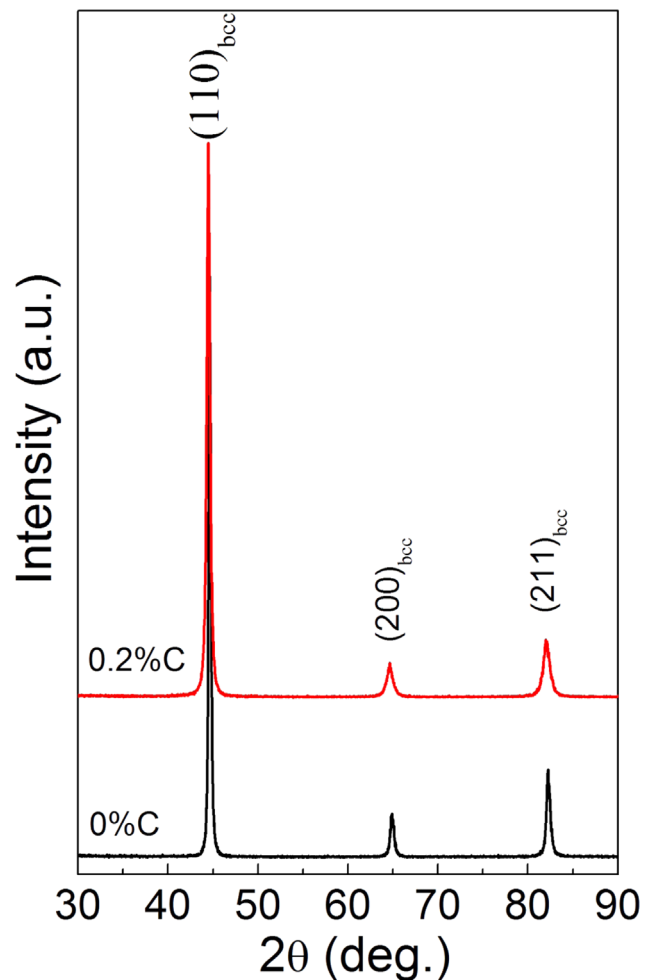

Fig. 1. X-Ray diffraction profiles of quenched pure iron and low carbon steel with 0.2%C.

black structures have long been considered as high density of dislocations [1,15]. In the present TEM observations, no distinct evidence of high density dislocations was observed. Actually, upon tilting the TEM specimen the contrasts of the bright blocks and dark blocks can be varied considerably, suggesting that these dotted black structures could not be ascribed to high density dislocations. Further, under proper tilting of the TEM specimen, twinned structures (as shown in Fig. 2(a)) emerge in some martensite variants. Thus, it is reasonable to conclude that twins, rather than high density dislocations, might be the substructure of the lath martensite in low carbon steels of the present study. As a matter of fact, high density dislocations were only observed in the martensite with ultralow carbon content such as 0.0026%C [2]. There is no convincing evidence to ascribe the dark contrasts of martensite blocks observed in early TEM studies to be high density dislocations.

In order to probe the detailed substructures inside the lath martensites, a further TEM characterization was carried out on the lath martensite variants. Fig. 3 shows the dark-field TEM micrograph and the corresponding selected area electron diffraction (SAED) patterns of a typical lath martensite. According to the bright field micrograph of twins as shown in Fig. 3(a) and the corresponding SAED pattern shown in Fig. 3(b), these twins are recognized to be {112} < 111> type, which is the most popular twin structure in bcc metals and alloys. This observation suggests that twins might be involved in lath martensite in low carbon steels. As for the contrast arising from dotted microstructures in
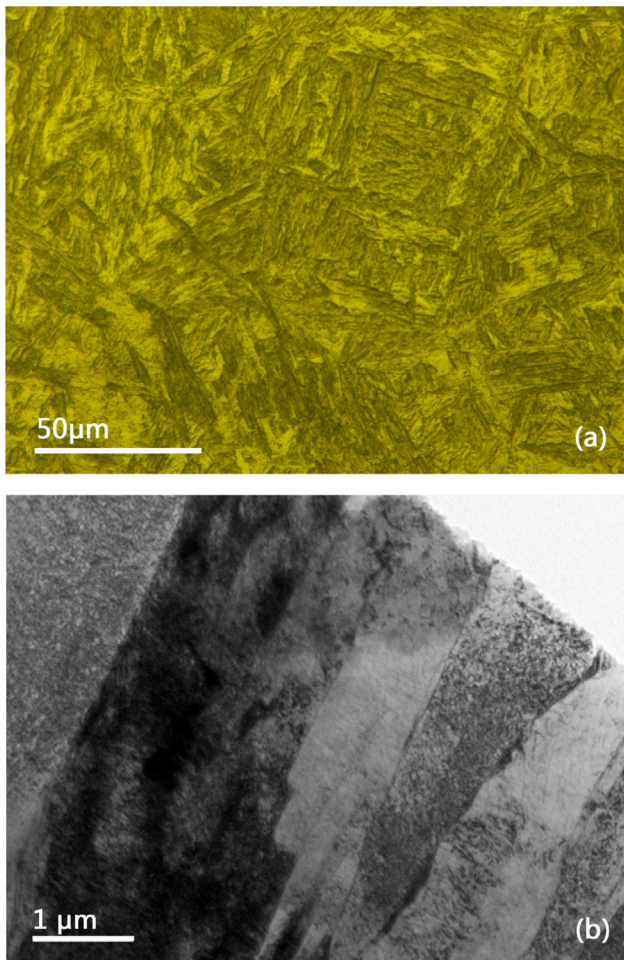
Fig. 2. Optical micrograph (a) and bright-field TEM micrograph (b) of the quenched specimens of low carbon steel with 0.2 wt%.

block of martensites, the present authors speculate that it might be resulting from twins under specific view of observation, e.g. the incident electron beam parallels the length direction of twins. Since the width of twins is in the order of nanometer scale, the ends of the twin columns could exhibit dotted structure when observing the twins along their length direction. Up to date, the prevailing recognition on martensite morphology deems the high density of dislocations to be the substructure of low carbon martensite. Nevertheless, some internally twined martensites have been observed in early investigation on low carbon steels with 0.14–0.34%C [17], whereas the dislocations or tangled dislocations martensites were observed only in steels with extra-low carbon content, for example, 0.0026%C and 0.0004%C [2,18].

Fig. 3(b) shows the $[011]_{bcc}$ zone SAED pattern in which the diffraction spots from bcc matrix and the conjugate spots from twins are indicated by squares of write solid line and dashed line, respectively. Fig. 3(c) is the dark field TEM micrograph imaged using the diffraction spot 1 outlined by the white circle in Fig. 3(b). It clearly reveals typical twin structures as well as the interfaces between the matrix and twins. Interestingly, in addition to diffraction spots from the matrix and twins, two extra spots can be found at $1/3(\bar{2}\bar{1}1)$ and $2/3(\bar{2}\bar{1}1)$. In order to determine the origin of the two extra spots, a dark field TEM micrograph was imaged using the spot 2 at $1/3(\bar{2}\bar{1}1)$, as shown in Fig. 3(d). By comparison with Fig. 3(c), some bright ultra-fine nanoscale particles can be observed lying at the interface between twins and matrixes. Note that these extra diffraction spots cannot be identified as any well-known carbide such as θ-Fe_3C or ε-$Fe_{2.4}C$. As a matter of fact, these

spots could be indexed as ω phase with primitive hexagonal structure, which was observed recently in the martensites in medium and high carbon steels [19]. As a metastable phase frequently appeared in Group IV metals and their alloys, ω phase usually precipitate accompanying the matrix in bcc structured metals or alloys, such as β-type Ti (Zr or Hf) alloys, β-brass, Ta, Mo and some Fe-based high-alloys, particularly when the bcc lattice becomes unstable by doping, high speed impact or high pressure [20–23]. The ω phase is always coherent with the bcc matrix with the lattice relationships of $a_\omega = \sqrt{2} \times a_{bcc}$ and $c_\omega = \sqrt{3}/2 \times a_{bcc}$. Accordingly, the results of the present study indcate the presence of ω phase in the lath martensites of low carbon steel, suggesting that the precipitation of ω phase might be a common feature of martensite in carbon steels.

In view of the fact that the ω phase always precipitate at the interface of bcc matrixes and twins in martensites, we can reasonably speculate a cooperative relation between the ω phase and the twins in martensite, irrespective of steels with high, medium and low carbon content. Referring to the whole picture of the lath martensites in low carbon steel given in Fig. 3, we can conclude that the lath martensite formed in low carbon steel of the present study is a composite structure, rather than a single phase of bcc solution with supersaturated carbon atoms.

Fig. 4 presents the high-resolution TEM micrograph of the $\{112\} < 111>$ twins in Fig. 3(a) and the corresponding fast Fourier transformed (FFT) diffraction pattern. In the high-resolution micrograph some nanometer sized particles outlined by white dashed lines are clearly observable at the twin boundaries and coherent with the matrix. The fast Fourier transformed (FFT) diffraction patterns taking from yellow square area confirm that these nanoparticles are ω phase with primitive hexagonal structure.

Numerous experimental studies have demonstrated that ω phase has a substantial influence on the mechanical properties of alloys such as Ti(Zr, Hf) alloys due to the hard and brittle nature of ω phase. In general, precipitates of ω phase could result in a significant strengthening effect on the alloys, and large volume fraction of ω particles could remarkably deteriorate the ductility of the alloys [24]. In light of the cooperative relationship between ω particles and twins in the martensites of carbon steels, it is reasonable to believe that the nanoscale ω particles cohered with the ferrite matrix could exert substantial influence on the motion of the twins, and then the mechanical properties of martensitic steels.

Very recently, Wu el al. proposed a mechanism for the formation of $\{112\} < 111>$ twins in bcc metals and alloys, i.e. ω-lattice mechanism [25]. According to this mechanism, the formation of $\{112\}<111>$ twin structure is closely related to the evolution of ω phase, i.e. the reverse transformation of ω to bcc lattice. As a matter of fact, the formation of metastable ω phase is always attributed to some aspects which caused instability of bcc metals and alloys, such as shock, internal stress and alloying elements [26]. As a result, the formation of ω phase in the low carbon steel indicates the reasonability for the presence of twins in martensite. Since the formation of ω in martensite is associated with the lattice instability caused by the alloying carbon in carbon steels [27], clarifying the dependence of martensite structures on the carbon content of steels could be helpful for understanding the mechanism of martensitic transformation in steels.

4. Conclusions

In the present study, an intensive TEM investigation was made to characterize the microstructures of the lath martensite in a low carbon steel of 0.2 wt%C, and the corresponding conclusions can be extracted as follows:
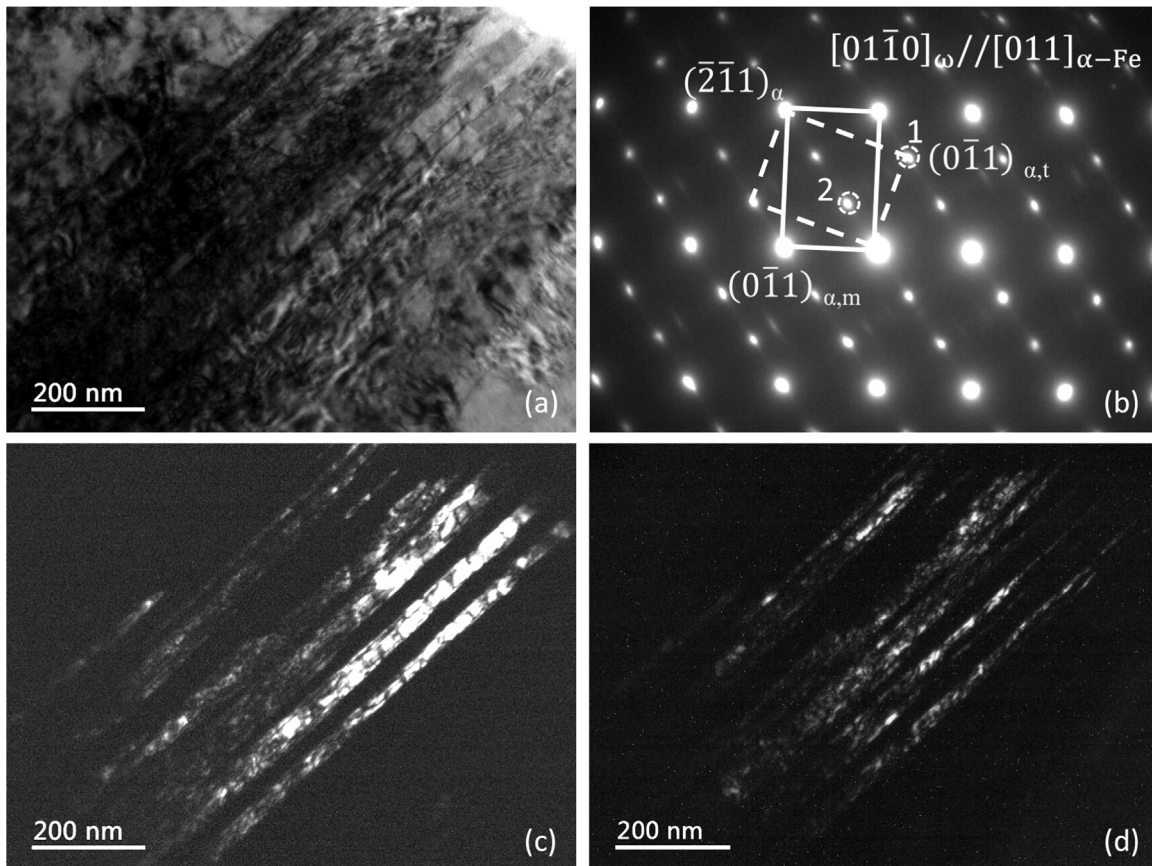
Fig. 3. Bright-field TEM micrographs (a) and SAED pattern (b) of the quenched specimen. (c) and (d) are the dark-field micrographs corresponding to the spot 1 and spot 2 shown in (b), respectively.
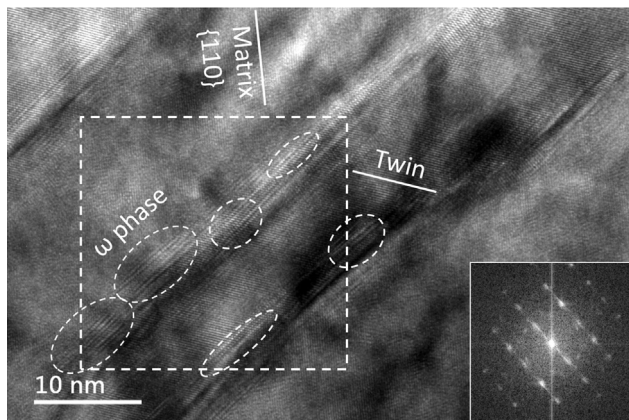

Fig. 4. High resolution transmission electron micrograph of twin boundaries in martensite. Insert: the corresponding fast Fourier transformed (FFT) diffraction pattern from the region in yellow square.

Twins, rather than high density dislocations, are the substructure of the lath martensites in the low carbon steel of 0.2 wt% C. Primitive hexagonal structured nanoscale ω precipitates cohering with ferrite matrix were observed at the twin boundaries of lath martensites.

Acknowledgments

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