A Simple Method for Observing ω -Fe Electron Diffraction Spots from $\langle 112 \rangle_{\alpha\text{-Fe}}$ Directions of Quenched Fe–C Twinned Martensite

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In twinned martensite, a metastable hexagonal ω -Fe phase always exists in the twin boundary region of the body-centered cubic (bcc) $\{112\}\langle 111 \rangle$ -type twin. The ω -Fe electron diffraction spots at the $1/3\{112\}_{\alpha\text{-Fe}}$ and $2/3\{112\}_{\alpha\text{-Fe}}$ positions have been treated as the twinning double diffraction effect previously. The ω -Fe spots fully cover the spots of the bcc matrix, twin and their double diffraction. Due to this, it is difficult to practically distinguish the ω -Fe diffraction spots from the sum of matrix + twin + double diffraction. Here, a simple method for observing the ω -Fe spots is introduced based on the twinning crystallographic analysis.

In this method, at first a $[011]$ zone axis is found in twinned martensite, containing the diffraction spots of twin and ω -Fe (previously double diffraction spots). It is then confirmed that the twin plane is inclined to the incident electron beam by means of the dark field observation. The reciprocal $\langle 222 \rangle^*$ direction (containing spots at $1/3\{222\}$ and $2/3\{222\}$ positions), is noted. A tilting is then performed keeping this direction un-tilted, i.e., tilting about this direction, to $\langle 112 \rangle$ zone axis. This requires about 30° tilting. If the ω -Fe spots are absent at the $1/3\{222\}$ and $2/3\{222\}$ positions when the zone axis reaches $\langle 112 \rangle$, then an opposite tilt is performed (since there are two tilting directions: clockwise and counter-clockwise), then, the ω -Fe diffraction spots can be seen at the $1/3\{222\}$ and $2/3\{222\}$ positions. A large twinned martensite at the TEM specimen edge is better for tilting to avoid any overlapping.

KEY WORDS: carbon steel; martensite; electron diffraction; twin; TEM.

1. Introduction

The body-centered cubic (bcc) $\{112\}\langle 111 \rangle$ -type twin is commonly recognized as the martensitic substructure in high carbon steels.^{1–10)} It was suggested that low M_s (martensite start temperature) favors the formation of the twinned structure.^{1,11–13)} In Fe–C system, low M_s corresponds to the alloy with high carbon content. Such a suggestion matches well with the experimental observations in high carbon alloys in which the twinning structure is the only martensitic substructure. Later, in low carbon alloys (~ 0.3 wt.%C), researchers found that an increase in M_s temperature by adding alloying elements did not decrease the propensity for transformation twinning, and it was concluded that M_s is not the sole factor determining twinning.¹⁴⁾ Our recent investigation results have confirmed that the twin formation is also strongly related to the austenitizing temperature and quenching conditions, and the twin could be commonly observed to be martensitic substructure in quenched martensite of low

carbon Fe-0.1C alloys.¹⁵⁾

The twin structure not only relates to the martensitic substructure or martensitic transformation, it is also directly related to the yield phenomenon of pure iron.¹⁶⁾ During yielding, the twinning seems to be easily formed in the carburized pure iron crystal, and therefore it was suggested that the small amount of carbon can favor the $\{112\}\langle 111 \rangle$ -type twinning in iron materials.¹⁶⁾ It seems that the role of the twin in affecting the microstructure and mechanical properties is complex, and even the formation mechanism itself of the transformation twin is still unclear.

A recently proposed martensitic transformation model has explained that the twinning structure can automatically form during the $\gamma\text{-Fe} \rightarrow \alpha\text{-Fe}$ transition, and the twin can actually be treated as the martensitic transformation product regardless of the carbon content.^{15,17)}

In twinned martensite, the presumed double electron diffraction spots at $1/3\{112\}$ and $2/3\{112\}$ are always observed together with the twinned structure during transmission electron microscopy (TEM) observations.^{6–10)} The nature of these spots at $1/3\{112\}$ and $2/3\{112\}$ have been recently explained based on a new metastable hexagonal

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ω -Fe phase (AB2-type, space group: $p6/mmm$, 3 atoms in one unit cell), which is distributed at the twin boundaries and has the crystallographic relationship with α -Fe: $a_\omega = \sqrt{2} \times a_\alpha$, $c_\omega = \sqrt{3}/2 \times a_\alpha$, and orientation relationship: $[011]_{bcc} // [2\bar{1}\bar{1}0]_\omega$, $[\bar{1}13]_{bcc} // [2\bar{1}\bar{1}3]_\omega$, $[120]_{bcc} // [2\bar{1}\bar{1}6]_\omega$, $(\bar{2}1\bar{1})_{bcc} // (0\bar{1}10)_\omega$.^{18–21)} Theoretical calculation results have also predicted the existence of the metastable ω -Fe phase and its relationship with the twin boundary (the twin boundary structure and the ω -Fe phase can stabilize each other energetically).^{21–25)} The ω -Fe phase has a particle-like morphology, the size is about 1–3 nm and the size distribution is very narrow.²⁰⁾

In this paper, a detailed analysis of electron diffraction patterns during TEM observations has been carried out on the $\langle 110 \rangle_{\alpha\text{-Fe}}$ zone axes of the twinned martensite formed in Fe–C binary alloys. The purpose is to explore a simple method to obtain the ω -Fe electron diffraction spots during TEM observations. In case of the bcc $\{112\}\langle 111 \rangle$ -type twinning system, no double diffraction should appear theoretically when the incident electron beam direction is perpendicular to the twinning plane. Nonetheless, some extra diffraction spots at $1/3\{222\}$ and $2/3\{222\}$ positions can actually be observed in twinned martensite, and these extra spots must be from a second phase. In the present investigation, a simple method will be explained to find the $\langle 112 \rangle$ zone axis with ω -Fe diffraction spots in the twinned martensite.

2. Experimental Procedure

Ingots of alloys Fe-0.1C, Fe-0.2C, Fe-0.4C, Fe-0.6C, Fe-0.8C, Fe-1.0C, Fe-1.4C, Fe-1.6C and Fe-2.0C (wt.%) were prepared in a high vacuum induction furnace under Ar atmosphere. The ingots were solution treated at about 1200°C for 2 h and then hot forged into a thick plate with a thickness of about 20 mm. Small plate-like samples of size of about 10 mm $\times$ 10 mm $\times$ 1.5 mm were cut from the 20 mm thick plates. The small sample plates were then put directly into a furnace at various austenitized temperatures with Ar gas flow for 30 min, and then water-quenched.

To prepare thin foil specimens for transmission electron microscope (TEM) observations, the water-quenched samples were first thinned to about 50 μm thickness by mechanical grinding and polishing. Discs with a diameter of 3 mm were then punched from these samples and subjected to electro-polishing in a twin-jet electropolisher with a chemical solution of 10% HClO_4 and 90% ethanol at -30°C . An ion-mill (Fischione Model 1050 TEM Mill) was also used for cleaning the specimen surface at 4 kV for 5 min. Some specimens for TEM observations were directly thinned by ion-milling after mechanical grinding. No observable difference could be noticed between the samples prepared by these two methods. The microstructural observation was carried out on the TEM JEM 2000FX operated at 200 kV.

3. Results and Discussion

3.1. $\langle 011 \rangle$ Electron Diffraction Patterns in Twinned Martensite

In the water quenched Fe-0.8C (wt.%) alloy after austenitizing at 950°C for 30 minutes, all the martensite had the $\{112\}\langle 111 \rangle$ -type twinning structure as its substructure.

Figure 1 shows a typical twinned structure and the corresponding electron diffraction pattern. Twinned crystal contrast can be clearly seen in the dark field image (Fig. 1(b)) obtained using the $(01\bar{1})_{\text{twin}}$ diffraction spot. This contrast and the electron diffraction pattern reveal that the twinning plane is parallel to the incident electron beam and also parallel to the $[011]$ directions of both the matrix crystal and the twinned crystal.

The bcc $\{112\}\langle 111 \rangle$ -type transformation twins have been observed in all quenched martensite in the binary Fe–C alloys with various carbon contents (from 0.1 to 2.0) in the present investigation. Thus, in the present paper, representative TEM images from selected samples are shown. The twinning morphology (twin length and width) dependence on the various carbon contents will not be discussed here since the present article is mainly about the electron diffraction patterns of martensite.

Figure 2 shows TEM observation results from the water quenched Fe-1.0C (wt.%) sample after austenitizing at 1200°C for 30 minutes. Interestingly, the typical twinned crystal contrast could not be seen in any dark field image obtained using various diffraction spots. No twin boundary contrast can be seen in the dark field images even at a high magnification. Thus it can be concluded that the twinning plane is inclined to the incident electron beam, even though the two diffraction patterns of Figs. 1(c) and 2(b) are from the same zone axis $\langle 110 \rangle_{\alpha\text{-Fe}}$ parallel to the incident electron beam. These features can be observed in any twinned martensite in the quenched samples with various carbon contents.

Fig. 1. TEM observations on a twinned martensite in the as-quenched Fe-0.8C (wt.%) sample. (a) A bright field image. (b) A dark field image taken from the diffraction spot $01\bar{1}_{\text{twin}}$ in (c). (c) A SAED pattern obtained from the twinned martensite in (a). (d) The index of the diffraction pattern in (c). Along this observation direction, the twinning plane is parallel to the incident electron beam. Some extra weak diffraction spots in (c) may be caused by the TEM specimen surface oxide or re-deposition during the ion-milling process. (Online version in color.)

Fig. 2. TEM observations on a twinned martensite in the as-quenched high carbon (Fe-1.0C (wt.%)) sample water-quenched from 1 200°C. (a) A bright field image. (b) SAED pattern obtained from the dark contrast region in (a). (c)–(e) are the dark field images obtained from the spots indicated by the dashed arrows. (f) The index of the SAED pattern in (b). (g) Schematic illustration of the twinning plane, which is inclined to the incident electron beam or the $[110]$ direction indicated by a red arrow.

3.2. $\langle 011 \rangle$ Zone Axes Analysis of the $\{\bar{1}12\}\langle 1\bar{1}1 \rangle$ -type Twin

Figure 3 shows an atomic structure model of the $\{\bar{1}12\}\langle 1\bar{1}1 \rangle$ -type twinning structure. The matrix crystal and the twinned crystal are indicated in blue and orange color, respectively. The $[110]$ directions of both the matrix crystal and the twinned crystal are parallel to the $(\bar{1}12)$ twinning plane and perpendicular to the paper plane, *i.e.*, the $[110]$ directions from both the crystals are parallel to the incident electron beam during TEM observations. Such twinned structure has only one situation that can satisfy the following: both the $\langle 011 \rangle$ directions from the matrix and the $\langle 011 \rangle$ directions from the twin are parallel to the twinning plane $(\bar{1}12)$ and the incident electron beam direction. There are twelve possible directions for $\langle 011 \rangle$ zone axes as listed in Table 1, with 6 unique directions and their opposites.

The six directions listed in Table 1 are considered to the matrix crystal with possible parallel directions from the twinned crystal according to the crystallographic relationship shown in Fig. 3. It can be seen that there are six pairs of parallel matrix and twin directions, (1) $[011]_m/[101]_t$, (2) $[0\bar{1}1]_m/[141]_t$, (3) $[101]_m/[0\bar{1}1]_t$, (4) $[10\bar{1}]_m/[411]_t$, (5) $[110]_m/[114]_t$, and (6) $[1\bar{1}0]_m/[1\bar{1}0]_t$. The result shown in Fig. 1 corresponds to the case (6), while the results shown in Fig. 2 correspond to the cases (1) and (3).

In case (6), the twinning plane contrast will be visible, whereas it will be invisible in the other five conditions since the twinning plane will be inclined to the incident electron

Fig. 3. An atomic structural model of a bcc $\{\bar{1}12\}\langle 1\bar{1}1 \rangle$ -type twin. The twinning boundary plane $(\bar{1}12)$ is indicated by a horizontal dashed line, the crystal above this line is treated as the matrix crystal (blue color), and the one below this line is twin (orange color). The dots are in the same layer; while the open circles are in the upper or lower layer.

beam. In three of these cases, $\langle 114 \rangle$ direction of the twinned crystal will be parallel to the $\langle 110 \rangle$ direction of the matrix crystal. Figure 4 shows that in such cases the diffraction spots from the $[114]$ zone axis are fully overlapped by the spots from the $[110]$ zone axis. Double diffraction effect between these two directions will never produce any

extra diffraction spots in the composite diffraction pattern. Indeed, there are some $\langle 110 \rangle_{\alpha\text{-Fe}}$ directions which never show any twinned diffraction spots and the so-called double diffraction spots experimentally. These $\langle 110 \rangle_{\alpha\text{-Fe}}$ directions must match with the direction explained in Fig. 4.

Correspondences of these directions and planes can immediately be seen from pole figures. **Figure 5** shows

Table 1. All possible $\langle 011 \rangle$ directions in matrix crystal and the corresponding parallel directions in the twin crystal in a bcc $\{112\} \langle 111 \rangle$ -type twinning system.

Case	$\langle 011 \rangle$ directions	Direction in matrix crystal	The parallel direction in twinned crystal
(1)	$[011]$ $[0\bar{1}\bar{1}]$	$[011]$	$[\bar{1}01]$
(2)	$[0\bar{1}1]$ $[01\bar{1}]$	$[0\bar{1}1]$	$[\bar{1}41]$
(3)	$[101]$ $[\bar{1}0\bar{1}]$	$[101]$	$[0\bar{1}1]$
(4)	$[10\bar{1}]$ $[\bar{1}01]$	$[10\bar{1}]$	$[411]$
(5)	$[110]$ $[\bar{1}\bar{1}0]$	$[110]$	$[114]$
(6)	$[1\bar{1}0]$ $[\bar{1}10]$	$[1\bar{1}0]$	$[1\bar{1}0]$

a stereogram of matrix crystal (blue color in Fig. 5(a)) and twinned crystal (orange color in Fig. 5(b)) with $\{1\bar{1}2\} \langle \bar{1}11 \rangle$ -twinning relationship. The $\{112\}$ twinning plane is indicated. Only $[110]_m$ and $[110]_t$ directions can lie in the $(1\bar{1}2)$ twinning plane. Two other pairs of directions ($[101]_m$ and $[01\bar{1}]_t$, $[01\bar{1}]_m$ and $[101]_t$), are inclined to the twinning plane. The other $\langle 110 \rangle$ directions of the matrix crystal will be parallel to the corresponding $\langle 114 \rangle_t$ directions, all of which are inclined to the twinning plane. This crystallographic analysis is independent of the twin crystal size.

Fig. 4. Schematic electron diffraction pattern: (a) bcc $[011]$ zone axis, (b) bcc $[114]$ zone axis, and (c) the overlapped pattern of (a) and (b). The open dashed circles in (c) correspond to the diffraction spots in (b).

Fig. 5. Schematic representation of stereographic projection: (a) bcc (110) matrix crystal (blue color), (b) bcc (110) twin crystal (orange color), and (c) the $\{1\bar{1}2\} \langle \bar{1}11 \rangle$ -twinned crystals.

3.3. Tilting to Obtain ω -Fe Diffraction Spots

Based on the above analysis, there are three possible conditions for the twinning double diffraction effect to produce extra diffraction spots in the observed electron diffraction patterns, that is the cases (1), (3) and (6) listed in Table 1. In the case (6), and as shown in Fig. 1(c), it is generally impossible to tilt to the $\langle 112 \rangle$ axis perpendicular to the twinning plane in TEM since this tilting requires a large tilt of 90° . However, such a tilting can be carried out from the zone axis shown in Fig. 2(b) (cases (1) and (3) in Table 1), by tilting about a reciprocal direction $\langle 111 \rangle^*$ which contains ω -Fe diffraction spots at $1/3(222)_{\alpha\text{-Fe}}$ and $2/3(222)_{\alpha\text{-Fe}}$ positions. In this case tilting to zone axis $\langle 112 \rangle$ requires a tilt of about 30° . Such a simple tilting experiment is illustrated by a diagram in Fig. 6. Figure 6(a) is a schematic of the electron diffraction pattern of Fig. 2(b). Here, the dynamic diffraction effect is not considered; thus, the spots represent the diffraction spot positions. While tilting, the reciprocal axis $\langle 111 \rangle^*$ is kept in position (to watch for the diffraction effects in the reciprocal spots along it). By changing the tilt-

ing angle, the diffraction pattern (zone axis $\langle 112 \rangle$) shown in Fig. 6(c) can be obtained.

Figure 7 shows the experimental diffraction patterns and bright field images from the tilting suggested in Fig. 6. Figures 7(a) and 7(b) show a bright field TEM image and the corresponding diffraction pattern from martensite in water-quenched Fe-0.8C (wt.%) sample after austenitizing at 950°C for 30 minutes. Dark field observations in this condition did not show any twinning contrast. Thus, this diffraction condition is similar to that shown in Fig. 2, in which the twinning plane is inclined to the incident electron beam. This condition corresponds to the position marked 'O' in Fig. 5(c). Tilting was then performed about the $\langle 111 \rangle^*$ axis (indicated by an orange line) of the diffraction pattern in Fig. 7(b). Comparing the diffraction pattern in Figs. 7(c) to 7(e), a tilt of about $\sim 25^\circ$, a diffraction spot 011_α (indicated by a white arrow) appears. Further tilting led to the expected $\langle 112 \rangle$ zone axis, as shown in Fig. 7(g). An important point is that the diffraction spots along $\langle 111 \rangle^*$ reciprocal direction never disappeared during the whole tilting. The 0001_ω and 0002_ω diffraction spots should be clearly visible in the zone axis $\langle 112 \rangle$ marked 'A' in Fig. 5(c), thus, the purpose of the tilting is to reach the axis A position where two $\langle 112 \rangle$ directions are parallel to each other. However, tilting in the opposite direction will lead to another $\langle 112 \rangle$ axis marked 'B' in Fig. 5(c), which is also inclined to the twinning plane but without $\{0001\}_\omega$ and $\{0002\}_\omega$ spots. These experimental results clearly show that $\{0001\}_\omega$ and $\{0002\}_\omega$ spots are not due to the double diffraction effect in the observed $\langle 112 \rangle$ directions (Fig. 7(g)).

For keeping the original diffraction patterns, the indexing of diffraction spots was not made in Fig. 7, the detailed index can be referred to Fig. 6. Though this tilting experiment was performed on the martensite in the water-quenched Fe-0.8C (wt.%) sample after austenitizing at 950°C for 30 minutes, one can perform this tilting experiment on any twinned martensite. However, larger martensite grain or structure would

Fig. 6. Schematic electron diffraction pattern: (a) the $[2\bar{1}\bar{1}0]_{\omega\text{-Fe}}/[011]_{\alpha\text{-Fe}}$ zone axes, (b) the $[2\bar{1}\bar{1}0]_{\omega\text{-Fe}}/[011]_{\alpha\text{-Fe}}$ zone axes, the orange line indicates one of the $\langle 111 \rangle$ directions in bcc structure, and (c) the $[1\bar{1}00]_{\omega\text{-Fe}}/[2\bar{1}\bar{1}]_{\alpha\text{-Fe}}$ zone axes after (a) was tilted about the orange line in (b). (Online version in color.)

Fig. 7. TEM results from a water-quenched Fe-0.8C (wt.%) sample after austenitized at 950°C for 30 minutes: (a) bright field image revealing martensite morphology, (b) the corresponding SAED pattern obtained from the outlined region in (a), (c) SAED pattern after tilting $\sim 17^\circ$ about the orange line from (b), (d) the corresponding bright field image of (c), (e) SAED pattern after tilting $\sim 5^\circ$ from (c), (f) SAED pattern after tilting $\sim 3^\circ$ from (e), (g) SAED pattern after tilting $\sim 5^\circ$ from (f), (h) the corresponding bright field image of (g). (Online version in color.)

be better for carrying out the tilting in order to avoid too many diffraction spots coming from unnecessary grains if the grain size is too small for the selective aperture in TEM.

Based on the pole figure (Fig. 5(c)) of the twin, there is a short way for tilting to reach the axis at 'A' position if the tilting experiment was started from $\langle 111 \rangle_{\alpha\text{-Fe}}$ zone axes. One can see that the $[1\bar{1}1]_{\alpha\text{-Fe}}$ and $[\bar{1}1\bar{1}]_{\alpha\text{-Fe}}$ axes are close ($\sim 20^\circ$ tilting angle) to the 'A' position. Such experiment observation has been carried out and reported in the previous work.²¹⁾ However, it is difficult to distinguish the observed $\langle 111 \rangle$ zone axis whether it is the $[1\bar{1}1]_{\alpha\text{-Fe}}$ or $[\bar{1}1\bar{1}]_{\alpha\text{-Fe}}$ axis since all $\langle 111 \rangle_{\alpha\text{-Fe}}$ shows the exact same diffraction pattern.

A common concern that may arise here is about the uncovering of this ω -Fe phase so late. The following factors may be responsible for this recognition of the ω -Fe phase: (1) It is assumed that the twinning structure are commonly based on the bcc twin forms from bcc structure and fcc (face-centered cubic) twins form from fcc structure, while in fact the twinned martensite is formed directly from fcc austenite in quenched Fe–C alloys. (2) As explained earlier,^{15,18,20)} most of the ω -Fe diffraction spots are overlapped with bcc matrix and bcc twin, as well as the double diffraction spots, but were considered to be only double diffraction spots. (3) The size of the ω -Fe crystallites is too small to be resolved with clarity. The ultra-fine particle size (~ 1 nm) suggests that this phase may not be quite stable.

Based on the experimental observations and some theoretical considerations up to now, some remarks can be made as follows: the twin (bcc $\{112\}\langle 111 \rangle$ -type) does not exist without the ω -phase. The ω -Fe is not a very stable phase. In carbon steels, the carbon atoms can stabilize the ω -Fe, which, however, may not be enough. The twin boundary structure is also necessary to stabilize the ω -Fe phase. In turn, the ω -Fe can stabilize the twin structure. Thus all three — the ω -Fe phase, the carbon atoms or nitrogen atoms, and the twin boundary structure — are stabilizing each other. Once the twin disappears (detwinning), the ω -Fe together with carbon atoms will transform to carbides.

4. Conclusions

Electron diffraction analysis has been performed in Fe–C martensite to confirm the bcc $\{112\}\langle 111 \rangle$ -type twinning

and the underlying ω -Fe phase. $\langle 110 \rangle_{\text{matrix}}//\langle 110 \rangle_{\text{twin}}$ pair of axes have been distinguished from that parallel to twin plane and those inclined to the twin plane. In order to find the existing evidence of the metastable ω -Fe phase in the electron diffraction patterns from the twin, a simple experimental tilting method has been suggested and performed. By this method, the ω -Fe electron diffraction spots $0001_{\omega\text{-Fe}}$ and $0002_{\omega\text{-Fe}}$ along one of the $\langle 112 \rangle_{\alpha\text{-Fe}}$ directions of the twin can be definitely observed and identified unambiguously. This tilting can be practically carried out from the coincident $\langle 110 \rangle$ zone axes of the matrix and the twin, containing the ω -Fe diffraction spots, which is inclined to the twinning plane.

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