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Omega transition accompanied by mechanically-induced twinned martensite

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Abstract

We present here an analysis on omega transition process during martensitic transformation. Martensitic transformation occurred during deformation at room temperature, avoiding the influence of auto-tempering on metastable omega phase. Based on the crystallographic relationships of twinned crystals, the twin interface of twinned martensite was characterized by considering the effect of direction of incidence electron beam on diffraction patterns through pole figures. Omega phase only existed at the boundaries of twinned martensite with single variant. It was proposed that lattice invariant twin shear during the dynamic transformation of twinned martensite promoted the formation of omega phase.

1. Introduction

Omega (ω) phase is present as a metastable hexagonal structure in ordered body-centered cubic (bcc) alloy systems, especially in groups IV, V and VI transition metals and their alloys [1, 2]. The ω-lattice usually forms on pair of (111) planes within bcc-lattice, leaving the adjacent (111) planes unaltered [1]. In {112} (111) mechanical twinning system of bcc metals and alloys, transformation mechanism of ω phase was proposed where mechanical twinning shear dominated the formation of ω-phase in Ta-W-based alloys and Ti–Nb-based alloys [3–5]. The ω-lattice mechanism was proposed involving the possibility of reverse transformation from ω to bcc in Ti-based alloys [6].

The ω phase was first reported in Fe–C alloys (group VIII), especially in twinned martensite with high carbon content [7, 8]. The formation mechanism of two-phase structure (ω + bcc) assumed that austenite directly transformed to ω + bcc structures [9, 10]. Based on ω-lattice mechanism in bcc alloys, the formation mechanism of twinned and lath martensite was suggested [11]. Twinned martensite formed from the unstable ω phase through ω-lattice mechanism during the quenching process. Subsequently, twinned martensite transformed to lath martensite during auto-tempering via movement of twin boundaries [11–13]. Generally, the inevitable auto-tempering during quenching makes the ω phase unstable and leads to microstructure evolution of martensite. This makes it difficult to characterize the atom–scale structure of ω phase.

The transformation mechanism of ω phase has not been analyzed by combining with martensitic transformation process. In this study, martensitic transformation was induced by deformation at ambient temperature, which excluded the influence of auto-tempering on metastable ω phase. To characterize the twin interface of twinned martensite, the effect of direction of incidence electron beam on diffraction patterns was analyzed together with the pole figures based on the crystallographic relationships of twinned crystals. After selecting the right direction of incidence electron beam, the substructure of deformation induced martensite was characterized by high-resolution transmission electron microscopy (HRTEM). Results indicated that ω phase was embedded in the twin interface of deformation-induced twinned martensite with only one variant along the omega transition accompanied by mechanically-induced twinned martensite
It was suggested that $\omega$ phase was a by-product of twinned martensite and the shear of lattice-invariant twin deformation promoted $\omega$ transition in medium manganese steels.

2. Materials and methods

The actual chemical composition of steel was Fe-0.23C-5.65Mn-1Al (in wt.%). Cold-rolled sheets were first austenitized at 850 °C for 15 min and then water quenched to room temperature to obtain martensite. The quenched steels were subsequently intercritically annealed at 680 °C for 10 min, followed by quenching to room temperature. The tensile specimens of gage length 25 mm were prepared along the rolling direction according to the ASTM E8 standard. Tensile deformation was used to introduce pre-strain (engineering strain of 3%) into the annealed steel with a constant crosshead speed of $6.7 \times 10^{-4}$ s$^{-1}$.

X-ray diffraction (XRD) studies were carried out to determine the amount of austenite and the volume fraction calculated based on the model described in reference [14]. The average carbon concentration in austenite was obtained by relation [14]:

$$\alpha_{\gamma} = 3.556 + 0.0453x_C + 0.00095x_{Mn} + 0.0056x_{Al},$$

where $x_C$, $x_{Mn}$, and $x_{Al}$ are the concentration (wt%) of carbon, manganese, and aluminum in retained austenite, respectively. XRD experiments were conducted on a D/max2400X-ray diffractometer (operated at 56 kV, 182 mA) with Cu Kα radiation at room temperature and the samples were scanned over a 2θ range from 40° to 100° with a step size of 2°/min that included ferrite and austenite peaks. The microstructure was characterized by scanning electron microscope (SEM, Zeiss Ultra 55) and transmission electron microscope (TEM, FEI Tecnai G2F20). Both TEM and HRTEM images were acquired at an accelerating voltage of 200 kV. Electron backscatter diffraction (EBSD) (Symmetry®, Oxford Instruments, Oxford, UK) maps were obtained at a step size of 0.05 μm and analyzed using the MTEX (version 5.28) texture analysis MATLAB (version 2016b) toolbox. TEM foils were twin-jet polished (StruersTenuPol-5) at a voltage of 20 V in a solution containing 5% perchloric acid.

3. Results and discussion

3.1. Microstructure evolution

A complete martensite structure was obtained on quenching to room temperature from 850 °C, as shown in figure 1(a), and also implied by the XRD patterns presented in the inset of figure 1(a). After subsequent annealing at 680 °C for 10 min, a typical dual-phase microstructure of medium Mn steels consisting of retained austenite and ferrite was achieved (figure 1(b)). The XRD patterns in figure 1(b) indicated that the volume fraction of austenite was ~30% after the annealing process.

Figure 2 shows the crystallographic analyses after annealing. This includes phase map and the inverse pole figures (IPF) in the Z direction, which is normal to the rolling plane. Grain boundaries were defined where crystallographic misorientations exceeded 3° for the misorientation of martensite lath 2°–5°. After annealing at 680 °C for 10 min, the original martensite laths became lath-shaped ferritic grains, some of which coalesced to form large grains, as shown in figure 2(a). Both lath-type austenite and globular austenite can be seen in figure 2(b) and the austenite located in the same prior austenite had similar orientation. The lath-type austenite was usually located at original martensite lath, sub-block and block boundaries, whereas globular austenite
located at prior austenite grain boundaries and within packets. In figure 2(c), the phase boundaries between austenite and ferrite were associated with K-S or N-W orientation relationship (OR), which were highlighted red or yellow, respectively. The ratio of the length of K-S/N-W was \( \sim 1.75:1 \). The angular tolerance for this analysis was \( \pm 2.5^\circ \), which was based on the 5.26° difference between the K-S and N-W OR [15]. Figures 3(a)–(b) shows the engineering strain–stress curves of the sample after annealing and the XRD patterns of the sample before and after 3% pre-strain, respectively. A significant amount of austenite transformed to martensite during tensile deformation and the volume fraction of austenite decreased from 30% to 13.8% after 3% pre-strain.

**Figure 2.** (a) Overlay EBSD IPF-Z map of ferrite and austenite with low angle boundaries (with misorientations > 3°) after annealing. IPF-Z map of austenite (b) and austenite/ferrite boundaries that exhibit special orientation relationships are highlighted in red (K-S OR) or yellow (N-W OR) (c).

**Figure 3.** (a) The complete and 3% pre-strain engineering strain-stress curves of samples after annealing at 680 °C for 10 min and (b) XRD patterns of samples after annealing and 3% pre-strain.
The microstructure evolution after 3% pre-strain was further characterized by TEM. Figures 4(a)–(d) shows typical morphology of retained austenite after 3% pre-strain. After 3% pre-strain, in the bright field image (figure 4(a)), the retained austenite was feeble due to deformation, and streaks appeared in majority of the retained austenite. The selection area electron diffraction (SAED) pattern shown in figure 4(a) indicated that the streaks were stacking faults and no twins or epsilon-martensite were observed in the retained austenite. The dark-field image taken by the \( g = 002 \) \(<\gamma\>\) diffraction spot is shown in figure 4(b). The stacking faults in austenite were clearly seen in the austenite outlined by red broken line, which were the nucleation sites for martensitic transformation. Besides, some austenite partially transformed to dislocation martensite, with resulting microstructure consisting of austenite and dislocation martensite, as outlined by white broken line in figure 4.

The SAED pattern inserted in figure 4(c) reveals that \( \alpha' \) martensite formed in the deformed austenite and the OR between austenite and martensite followed K-S OR, \((111)_a//\langle101\rangle_m, [10\overline{1}]_a//\langle111\rangle_m\). The dark field image (figure 4(d)) taken using the \( g = 020 \) \(<\gamma\>\) diffraction spot shows that part of the austenite transformed to \( \alpha' \) martensite. A careful TEM tilting experiment did not find any twin contrast in the \( \alpha' \) martensite.

However, if the austenite completely transformed to martensite, twinned martensite formed. The TEM images of twinned martensite after 3% pre-strain are shown in figures 5(a)–(d). The SAED pattern along the \( [01\overline{1}]_{\text{bcc}} \) zone axis (figure 5(b)) reveals that the twin regions have a crystalline orientation of \((211)\langle1\overline{1}1\rangle\), where \((211)\) and \( [1\overline{1}1] \) are the plane and twinning direction, respectively. A dark field image of twins was obtained using the spot marked c, revealing a high density of twinned martensite of several nanometers (figure 5(c)). In addition to the diffraction spots of matrix and twin crystals, two other spots were also found along with the \((211)\) plane in the twinned martensite, as shown figure 5(b). The dark field image (figure 5(d)) obtained by the extra spot marked d showed that some bright ultra-fine nanoscale plates were located between the twinning planes. The crystals in this twinned martensite are most likely to be \( \omega \) structures, which are identical to twinned martensite [16]. The orientation relationships between martensite and \( \omega \) structure followed: \((211)_{\text{bcc}}//\langle10\overline{1}\rangle_m, [0\overline{1}1]_{\text{bcc}}//\langle01\overline{1}\rangle_m\). However, the extra spots may also be double diffraction spots of \( \{112\} \langle1\overline{1}1\rangle\)-type twinning.
structure. To avoid the effect of double diffraction on diffraction analysis, the selection of direction of incident electron beam during TEM characterization is discussed below.

### 3.2. Atomic-scale characterization of interface of twinned martensite

When diffraction patterns of \{112\}〈111〉-type twinned martensite are analyzed in bcc system, the commonly used low-order asymmetric diffraction patterns including \{112\} spots are those with zone axes 〈110〉 and 〈113〉. Figure 6 shows stereographic projection of crystal directions of matrix (triangle) and twinned crystal (dot) with (112)[111]-twinning relationship. The crystal direction and planes can be seen from the pole figures and the twinning plane 〈112〉 is indicated with black color. As indicated in figure 6(c), there are six crystal directions of the twinned crystal parallel to 〈110〉. Only three 〈110〉 directions can be parallel to the corresponding 〈110〉, which were confirmed as 〈110〉, 〈011〉, 〈011〉, and 〈101〉. When the direction of incident electron beam was parallel to these crystal direction pairs, the diffraction patterns were diffracted from both the matrix and twin crystals like figure 5(b). However, the nature of these diffraction patterns was different. Only pair 〈110〉 was parallel to the twinning plane (112), and small streaks of diffraction spots along (112) appeared because of intersection of elongated reciprocal rod and Ewald’s sphere in reciprocal space. On the other hand, the other two crystal directions pairs were inclined to the twinning plane, and the double diffraction pattern would appear easily because of overlapping of twinned crystals in the direction of the incident electron beam. Analyzing from the zone axes 〈113〉 in a similar manner, as shown in figure 6(d), there were six pairs of parallel crystal directions between the matrix and twinned crystal following 〈113〉, 〈313〉, 〈113〉, 〈131〉, 〈131〉, and 〈131〉, respectively. However, only the first two pairs were parallel to the twinning plane (112). Accordingly, whenever the direction of incident electron beam is parallel to zone axes 〈110〉 or 〈113〉, there is only one-third possibility to be parallel to the twinning plane. Therefore, directions of the incident electron beam should be carefully selected, especially during atomic-scale characterization, to avoid observing the overlapped structure of twinned crystals.
In our study, although the incident electron beam was parallel to the twinning plane as indicated by the streaking of diffraction spots in figure 5(b), the double diffraction may still occur because of fine twins and thickness of TEM samples [17]. To weaken the effect of thickness on double diffraction, a twinned martensite located around the hole of TEM sample was selected for characterization, as shown in figure 7. The streaking of diffraction spots in figure 7(b) indicated that the direction of incident electron beam parallel to \(\{131\}_m\) was also parallel to the twinning plane \(\{112\}\). This provided an ideal condition to characterize the twin interface of the twinned martensite. Figures 7(c) and (d) are dark field images obtained from \(g = 011\), and the diffraction spot marked by d, respectively.

The twin interface of the twinned martensite in figure 7 was further characterized by high-resolution transmission electron microscope. Figures 8(a)–(b) are HRTEM images showing the interface structure of \{211\} \(\{111\}\)-type twinned martensite. According to the analysis of electron diffraction in figure 7(b), the twin interface was perpendicular to the plane of paper and the matrix and twinned crystal were mirror-symmetric with respect to the twin interface. Figure 8(b) shows corresponding inverse fast Fourier transformed image which originated from the white broken-line region in figure 8(a). The large interplanar spacing in figure 8(b) suggested that the nanoscale particles embedded in the twin boundaries are most likely to be \(\omega\) phase. As shown in figure 8(b), the measured interplanar spacing of \((10\overline{1}0)_t\) and \((01\overline{1}1)_t\) planes are \(\sim 3.61\) Å and 2.04 Å, respectively. According to the diffraction analysis, the hexagonal \(\omega\) phase and its bcc matrix had a unique dependence of lattice parameters:

\[
a_{\omega} = \sqrt{2} a_{hcc}, \quad c_{\omega} = \frac{3\sqrt{2}}{2} a_{hcc}.
\]

In twinned martensite, the lattice parameter of \(\alpha\)-Fe was \(\sim 2.852\) Å and \(\omega\)-Fe lattice parameter was \(a_{\omega} = 4.033\) Å, \(c_{\omega} = 2.470\) Å [18]. Figure 8(d) shows the simulated electron diffraction pattern of primitive hexagonal lattice, which is in good agreement with the fast Fourier transformed diffraction pattern (figure 8(c)). Furthermore, the interplanar space \((10\overline{1}0)_t\) and \((01\overline{1}1)_t\) planes were calculated to be 3.493 Å and 2.017 Å, respectively, which were almost consistent with HRTEM results in figure 8(b).
Figure 7. (a) Bright field image of twinned martensite located around the hole of TEM sample. (b) The corresponding electron diffraction analysis from the selected area in (a). (c) and (d) are dark field images obtained from the spots in (b) marked as c and d, respectively.

Figure 8. HRTEM results of $\omega$ phase in twinned martensite. (a) The HRTEM micrographs of $\{112\}\langle 111 \rangle$-type twinned martensite, with the direction of incident electron beam parallel to $\langle 131 \rangle_m$. (b) Inverse fast Fourier transformed image of the square region outlined by white dash line in (a) showing twinned crystals and $\omega$ phase embedded at the twin interface. (c) The fast Fourier transformed image of square zone in (a). (d) Simulated electron diffraction pattern along the $(011)_{\omega}$ zone axis diffracted from primitive hexagonal lattice with parameters of $a_\omega = 4.033$ Å and $c_\omega = 2.470$ Å, where $a_\omega$ is the parameter for $x$-axis and $c_\omega$ is the parameter for $z$-axis.
It was confirmed by HRTEM that ω phase forms in mechanically-induced twinned martensite with single variant. But whether it pre-existed or was a by-product phase of martensitic transformation needed discussion. According to the structural difference between ω lattice and bcc lattice, if unstable ω phase reversed to bcc structure through ω-lattice mechanism, there was a 50% probability for pre-existing ω phase transforming to twinned-variant bcc structure [5]. Thus, if ω phase existed before martensitic transformation, there would be more twinned martensite plates after 3% pre-strain. However, it can be seen from figures 4 and 5 that only few austenite islands transformed into twinned martensite, and majority of retained austenite transformed to lath martensite after 3% pre-strain. Moreover, ω phase embedded in the twinned boundary had a single variant, which was inconsistent with the four variants of ω transformed directly from austenite [10]. Therefore, it is suggested that the ω phase in twinned martensite is not a phase that exists before martensitic transformation. The formation must be closely related to the transformation of internal twins.

3.3. ω transition induced by \{112\} (111) shear in twinned martensitic transformation

The process involved in the formation of twinned martensite can promote the formation of twin shear stress promoting ω transition. When martensitic transformation occurs at low temperature, the stress concentration at the tip of the martensite plate grows giving rise to the lattice invariant twinning shear in martensite [19]. It has been proposed that the twinned martensite forms by a dynamic process [20]. Figure 9 shows a schematic diagram of the formation of internal twinned martensite plate. The increased thickness of internal twin plate can induce inverse stress. To release the inverse stress, a crystal with the same orientation as the first twin plate is obtained in the same martensite plate. By repeating such process, thin internal twinned plates are consequently produced in the martensite plate. The local high stress between the twinned plates would then promote the formation of ω phase. The lattice correspondence of \{112\} (111)-type bcc twins and ω structures are shown in figure 9. As reported for bcc metals, the (111) shear in \{112\} plane plays a key role in ω phase transition [3–5, 21]. In Ti–Nb-based alloys, it was shown that there was a distinct energy barrier for β (bcc) to ω transition, which can be overcome by \{112\} (111) shear [4]. Detailed structural characterization at atomic scale also indicated that there was a kind of under-developing of ω transitional structure formed along the longitudinal twin boundaries, which provided the conclusive evidence for shear dominated mechanism [5]. Hsiung and Lasilla [3] also interpreted the internal twinning and stress-induced ω transition by dislocation mechanism. It was proved that the growth of omega phase was controlled by the movement of 1/3 (111) dislocation and was expected to be slower than the growth of twin domain that was controlled by the movement of 1/6 (111) dislocations. In our study, compared with the length of twin plates, the ω phase had a shorter length, as shown in figure 8, which was consistent with the dislocation mechanism. In addition, it has been shown that solid solution can increase shear stress and promote the increase of twins and ω phase in bcc metals [3, 4]. In this study, both the solid solution of manganese and the low transformation temperature at room temperature would promote the shear stress and decrease the thickness of twinned plates. The average carbon content in retained austenite have reached to ~0.61 wt.% according to XRD results. The carbon also promotes the twin shear stress of twinned martensite formation. The thickness of mechanically-induced twinned plate is fine ~5 nm. And the carbon could segregate to the twinned interface during the dynamic transformation of twinned martensite. The carbon segregation played two roles in promoting omega transition. It has been proved that the fulfill orientation could benefit the transition of omega phase [22]. The carbon segregation would decrease the lattice parameter of twinned plate. 

![Figure 9. Schematic illustration of the formation of plate like ω phase, which is promoted by twinning shear during dynamic transformation of twinned martensite.](image)
and make the orientation of martensite and omega more fulfilled. What’s more, based on the first-principles study on omega phase in steel [23], the carbon segregation into omega phase in the twinned interface would make the omega phase stable. On the contrary, in pure iron, although nano-twinned martensite could be obtained via high-pressure, ω structure did not form [24].

4. Conclusions

In this study, lath and twinned martensite were obtained at room temperature by deformation. The characterization of twin interface of twinned martensite using zone axes ⟨110⟩ or ⟨113⟩, suggested that there was only one-third possibility for the direction of incident electron beam to be parallel to the twinning plane. HRTEM results confirmed that ω phase was embedded at the interface of twinned martensite with single variant. The formation mechanism of ω phase was discussed by considering dynamic transformation process of twinned martensite, which suggested that twin shear was critical in assisting ω phase formation in steels.

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Conflicts of interest

The authors declare no conflicts of interest.

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