Deformation behavior of duplex austenite and $\epsilon$-martensite high-Mn steel

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Received 13 August 2012
Accepted for publication 17 October 2012
Published 12 March 2013
Online at stacks.iop.org/STAM/14/014204

Abstract

Deformation and work hardening behavior of Fe–17Mn–0.02C steel containing $\epsilon$-martensite within the austenite matrix have been investigated by means of in situ microstructural observations and x-ray diffraction analysis. During deformation, the steel shows the deformation-induced transformation of austenite $\rightarrow \epsilon$-martensite $\rightarrow \alpha'$-martensite as well as the direct transformation of austenite $\rightarrow \alpha'$-martensite. Based on the calculation of changes in the fraction of each constituent phase, we found that the phase transformation of austenite $\rightarrow \epsilon$-martensite is more effective in work hardening than that of $\epsilon$-martensite $\rightarrow \alpha'$-martensite. Moreover, reverse transformation of $\epsilon$-martensite $\rightarrow$ austenite has also been observed during deformation. It originates from the formation of stacking faults within the deformed $\epsilon$-martensite, resulting in the formation of 6H-long periodic ordered structure.

Keywords: deformation-induced martensitic transformation, stacking faults, austenite, high-Mn steel

1. Introduction

Austenitic high-Mn steels have received a great deal of attention in recent years since they have an excellent combination of high tensile strength and ductility [1–3]. and the current interest in these steels has focused on improving their tensile properties for their possible use in automotive components [4]. It is expected that the use of these austenitic high-Mn steels could lead to significant weight reduction in structural and energy-absorbing components of automobiles. It is well known that the deformation behavior of austenitic high-Mn steels varies depending on the stacking fault energy (SFE), which is a function of alloy composition, e.g. Mn [5]. In general, deformation behavior becomes transformation-induced plasticity (TRIP) when the SFE is below 20 mJ m$^{-2}$, twinning-induced plasticity when the SFE is 20–40 mJ m$^{-2}$, shear-band-induced plasticity or microband-induced plasticity when the SFE is near 100 mJ m$^{-2}$ and slip when the SFE is above 100 mJ m$^{-2}$.

The microstructure of these austenitic steels can be made to contain ferrite, $\epsilon$-martensite, $\alpha'$-martensite and $\kappa$-carbide in various proportions depending on the heat treatment and alloy compositions [6–10] and therefore the development of high-Mn steels in various grades is possible. Among various second phases, $\epsilon$-martensite is of interest since it can undergo the deformation-induced phase transformation to $\alpha'$-martensite [4, 6, 11–13], possibly contributing to the TRIP effect, which might increase the work hardening rate. In addition, the austenite matrix itself can show the TRIP effect by deformation-induced phase transformation.
to ε-martensite. Therefore, steels consisting of duplex austenite and ε-martensite structures can exhibit multiple TRIP effects from deformation-induced phase transformation of austenite → ε-martensite as well as ε-martensite → α′-martensite. Tomota et al [6] conducted a fundamental study of the microstructural effect of various binary high-Mn steels on tensile behavior. It shows that for the steels containing Mn between 16 and 20 wt%, there is a deformation-induced phase transformation of austenite → ε-martensite, showing the occurrence of multiple deformation-induced martensitic transformation (DIMT). However, these steels showing the occurrence of multiple DIMT have a worse combination of tensile properties than the 25Mn binary steel showing only one DIMT, i.e. austenite → ε-martensite. Gaining a detailed understanding of the progress of DIMT with strain in duplex austenite and ε-martensite steels and its effect on tensile properties is crucial for the development of high-performance high-Mn steels. The objective of the present work is to investigate the evolution of DIMT as a function of applied strain and to elucidate how the DIMTs to ε-martensite and α′-martensite affect the tensile deformation behavior of the duplex austenite and ε-martensite steels. The detailed microstructural evolution with strain has been investigated by in situ transmission electron microscopy (TEM) and electron back-scattered diffraction (EBSD).

2. Experimental details

The steel used in this study has a nominal composition of Fe–17Mn–0.02C (wt%), which was prepared by induction melting in an Ar atmosphere. The analyzed composition is Fe–16.90Mn–0.018C and the concentrations of other elements were kept less than 100 ppm. The steel was soaked at 1150 °C for 2 h, followed by hot rolling to the final thickness of 31.8 mm (a total reduction of 55%) with a final rolling temperature of 850 °C. Specimens were cut from a one-fourth portion of the surface to avoid the central Mn segregation and were machined by the electrical-discharge method at room temperature to prevent any deformation during the specimen preparation process. Microstructure was observed by optical microscopy, electron backscattering diffraction (EBSD) and TEM. In order to distinguish each constituent phase in the optical micrograph, the electro-polished specimen was color-etched by using an etchant of 5% K2S2O7 and 95% distilled water for 10 s. For EBSD analysis, the surface of the polished specimen was electro-etched with an etchant of 5% perchloric acid and 95% acetic acid to remove the residual stress on the surface for a reasonable analysis. Real-time deformation behavior was observed by using in situ TEM with a cross head speed of 0.1 μm s⁻¹. Tensile tests were conducted in the longitudinal direction at 298 K using the flat specimens with a gauge length of 12.6 mm, a gauge width of 5 mm and a gauge thickness of 1 mm at the strain rate of 10⁻³ s⁻¹. X-ray diffraction (XRD) analysis was conducted by using a multi-purpose high-power x-ray diffractometer with Cu Kα radiation on the longitudinal section of the tensile specimen. A thin surface of each specimen was removed by electro-polishing to minimize the effects of surface damage and oxide layer on XRD results. Based on the XRD results, the changes in the phase fraction and the stacking fault probability (SFP) were calculated at the strain of 0, 0.02, 0.07, 0.12, 0.17 and 0.22.

3. Results

3.1. Microstructural changes during deformation

The typical microstructure of the steel before deformation is shown in figure 1(a), where dark brown and light brown colors correspond to austenite and ε-martensite, respectively. It shows that the phase fractions of austenite and ε-martensite are 55 and 45%, respectively, and austenite is surrounded by ε-martensite having the morphology of a plate [4]. After deformation, there is the formation of α′-martensite (dark blue) and additional ε-martensite. In addition, slight thickening and deformation of pre-existing ε-martensite plates are observed as shown in figure 1(b). The details of such microstructural changes with deformation can be more clearly seen in the results of EBSD analyses of the same area (figures 1(c)–(e)). They show that there is an initial transformation of austenite → ε-martensite followed by the transformation of ε-martensite → α′-martensite (see, e.g., the circled area). The transformation of austenite → ε-martensite occurs by either the formation of a new ε-martensite plate within the previously formed austenite or by the thickening of the pre-existing ε-martensite. It can also be noted that the transformation of ε-martensite → α′-martensite occurs within an ε-martensite plate or at the intersection of two ε-martensite plates. Another important observation is the occurrence of reverse transformation of ε-martensite into austenite (for example, see the boxed area in figures 1(c) and (d)).

The deformation-induced phase transformation behavior was investigated by in situ TEM observation, and the snapshot images are shown in figures 2(a)–(d), corresponding to initial, intermediate 1, intermediate 2 and final stages, respectively. At an early stage of loading, α′-martensite is formed at the intersection of two ε-martensite plates and the formation of a thin ε-martensite plate within austenite is also observed (figure 2(b)). With an increase in strain, there is a growth of α′-martensite, but the ε-martensite plate impinging on the ε-martensite plate having a newly formed α′-martensite appears to be decomposed, suggesting the occurrence of reverse transformation of ε-martensite to austenite (figure 2(c)). With a further increase in strain, it can be seen that there is the formation of new ε-martensite near the previously formed α′-martensite as well as a further decomposition of impinging ε-martensite as shown in figure 2(d). In addition to the formation of α′-martensite at the intersection of two ε-martensite plates as shown in figures 2(b) and 3(a), two other cases of α′-martensite formation were detected as shown in figures 3(b) and (c). In the case of the formation of α′-martensite at the intersection of two ε-martensite plates (figure 3(a)), it shows that α′-martensite has the orientation relationship, (0002)ε/[110]α′ and [2−1−10]ε/[−1−11]α′, with only ε1-martensite, in which it nucleates and grows, suggesting that the formation
Figure 1. Optical micrographs of the same area (a) before and (b) after deformation with the strain of 0.2, respectively (dark brown; austenite, light brown; $\varepsilon$-martensite, and dark blue; $\alpha'$-martensite), and EBSD phase maps of the microstructure with the strain of (c) 0, (d) 0.15 and (e) 0.28, respectively (red, austenite; green, $\varepsilon$-martensite; and yellow, $\alpha'$-martensite). The tensile loading direction is horizontal.

of $\alpha'$-martensite is triggered by the impingement of an $\varepsilon_2$-martensite plate onto $\varepsilon_1$-martensite. As shown in figure 3(b), it is also found that $\alpha'$-martensite is formed within the $\varepsilon$-martensite plate, without any observable interactions with the surrounding austenite or $\varepsilon$-martensite. In addition, the formation of $\alpha'$-martensite directly from austenite without the association of $\varepsilon$-martensite was observed particularly in areas near the fracture surface, suggesting the direct transformation of austenite into $\alpha'$-martensite at a high stress level, similar to the result of a previous investigation [14]. Therefore, there is not only a deformation-induced phase transformation of austenite $\rightarrow$ $\varepsilon$-martensite and later into $\alpha'$-martensite but also a direct transformation of austenite $\rightarrow$ $\alpha'$-martensite at a high stress level. In any case, the constituent phases, austenite, $\varepsilon$-martensite and $\alpha'$-martensite, have the combination of S-N and K-S orientation relationships before and after deformation such as $(111)_\gamma/(0002)_\varepsilon/(110)\alpha'$ and $[101]_\gamma/[111]_\varepsilon/[-20]_\varepsilon/[111]_\alpha'$ [11, 15].

The change in the phase fractions with deformation was calculated by XRD as shown in figure 4. It shows that the phase fraction of austenite decreases, while those of $\varepsilon$- and $\alpha'$-martensite increase with increasing strain up to 0.12, at which the phase fraction of $\varepsilon$-martensite reaches a maximum. It can also be noted that the transformation of austenite is mostly into $\varepsilon$-martensite and not $\alpha'$-martensite.

One interesting observation is that after the alloy is strained more than 0.12, the phase fraction of austenite increases up to the strain of 0.17, followed by a decrease with further increasing amount of strain. On the other hand, the phase fraction of $\varepsilon$-martensite decreases up to the strain of 0.17, followed by a small increase with further increasing amount of strain. It also shows that the phase fraction of $\alpha'$-martensite continuously increases with increasing strain. It is believed that such an increase in the phase fraction of austenite at the strain of 0.17 is due to the occurrence of reverse transformation of some $\varepsilon$-martensite plates back into austenite as shown in figure 1(d) (see, e.g., the boxed area).

3.2. Tensile properties

Figure 5(a) shows the engineering stress–strain curve of the steel. It shows that the steel has a yield strength of 322 MPa, a tensile strength of 695 MPa, uniform elongation of 24% and total elongation of 26%. As compared to fully austenitic 36Mn steel, it has a higher yield strength, possibly due to the presence of a rather large amount of $\varepsilon$-martensite [6]. The nano-indentation test of each constituent phase shows that the hardness of austenite and $\varepsilon$-martensite is $\sim$40 and $\sim$50 GPa, respectively, at the maximum depth of 1500 nm, indicating that $\varepsilon$-martensite is harder than
Figure 2. Snapshot images of *in situ* TEM micrographs corresponding to (a) initial, (b) intermediate 1, (c) intermediate 2 and (d) final deformation stages.

Figure 3. TEM micrographs showing the formation of $\alpha'$-martensite (a) at the intersection of two $\varepsilon$-martensite plates, (b) within a single $\varepsilon$-martensite plate and (c) within austenite without the formation of $\varepsilon$-martensite. Respective SAD patterns and dark field images of $\alpha'$-martensite are also shown.
austenite. Therefore, 17Mn–0.02C steel could show a higher yield strength than other austenite-base high-Mn steels due to the presence of ε-martensite during deformation. This is consistent with the results of a previous investigation showing that the yield strength of high-Mn steels increases with increasing volume fraction of ε-martensite within the austenite matrix [6]. To understand the details of deformation behavior, the normalized work hardening rate was calculated as shown in figure 5(b). The normalized work hardening rate indicates the incremental work hardening exponent and describes the persistence of work hardening during plastic deformation [16, 17]. It is represented by \( \frac{d \ln(\sigma)}{d \ln(\varepsilon)} \), where \( \sigma \) is the true stress and \( \varepsilon \) is the true strain. It shows that the normalized work hardening rate gradually increases with increasing deformation up to the strain of about 0.1, after which it gradually decreases.

4. Discussion

4.1. Deformation-induced transformation behavior

One interesting observation is that the steel shows the occurrence of reverse transformation of ε-martensite into austenite as shown in figure 1(d). Considering that the phase transformation between austenite and ε-martensite is closely related to the formation of stacking faults (SFs) [1, 14], investigation of the variation in SFP with deformation would lead to a better understanding of the mechanisms behind the reverse phase transformation occurring in the present steel. In general, there are two types of SF sequences, type 1 and type 2, in hcp structures as shown in figure 6(a). Houška et al [18] have shown that two types of faults can be produced by the growing together of two out-of-phase hcp lattices, and type 1 fault can also be formed by partial slip which converts A planes into C planes and B planes into A planes. Therefore, type 1 fault contains four planes of fcc stacking (ABABCA), while type 2 fault contains three planes of fcc stacking (ABABC). SFP of ε-martensite was calculated based on the whole profile pattern fitting method combined with the SF model proposed by Warren as shown below [19]:

\[
H - K = 3N : \frac{\partial A^2_L}{\partial L} = \frac{1}{D_a},
\]

\[
H - K = 3N \pm 1, L = \text{even}: -\frac{\partial A^2_L}{\partial L} = \frac{1}{D_a} + \frac{|L|d_{HKL}}{c^2}(3P_{\text{type1}} + 3P_{\text{type2}}),
\]

\[
H - K = 3N \pm 1, L = \text{odd}: -\frac{\partial A^2_L}{\partial L} = \frac{1}{D_a} + \frac{|L|d_{HKL}}{c^2}(3P_{\text{type1}} + 3P_{\text{type2}}),
\]

where \( H, K \) and \( L \) are Miller indices, \( N \) is an integer, \( L \) is Fourier length, \( A^2_L \) is Fourier coefficient, \( D_a \) is domain size, \( d_{HKL} \) is d-spacing, \( c \) is a lattice parameter and \( P \) is an SFP. In this analysis, the different dependence of \(-\frac{\partial A^2_L}{\partial L}\) Lorentzian peak broadening on \( P_{\text{type1}} \) and \( P_{\text{type2}} \) could be obtained through the whole pattern profile fitting method by considering the combinations of reflections if \( D_a \) can be assumed isotropic. As shown in figure 6(b), the SFP of type 1 SF is almost 0, indicating that type 1 SF does not form in ε-martensite of the present steel. On the other hand, the SFP of type 2 SF drastically increases at the strain of 0.12. As mentioned previously, type 2 SF contains three layers of fcc stacking sequence, and therefore it can be thought that the formation of such a stacking sequence is linked to the reverse transformation of hcp ε-martensite→fcc austenite. A sudden increase in the SFP at the strain of 0.12 is possibly due to the formation of such a stacking sequence in short-range order, which eventually develops into long-range order as the amount of strain increases, with a resultant increase in the phase fraction of austenite at the strain of 0.17 as shown in figure 4. To confirm the formation of SFs in ε-martensite responsible for the occurrence of reverse transformation, a detailed TEM analysis was conducted on the ε-martensite shown in figure 3(a). Figure 7(a) shows enlarged images of ε2-martensite plate shown in figure 3(a). It can be seen that ε2-martensite plate contains fine defects along the long direction of the plate. Selected area diffraction (SAD) analyses of ε2-martensite plate and its surroundings show that regions (i), (ii) and (iv) show the typical SAD patterns of the zone axes of \([1\bar{1}0]\)γ and \([2\bar{1} \bar{1}0]\)ε. However, the SAD pattern of region (iii) containing fine defects shows the presence of a 6H structure characterized by the presence of SFs in every sixth layer [20], which is the same as type 2 SF. This 6H structure is one of the long periodic ordered (LPO) structures found in several alloys. LPO structures such as 4H, 6H, 9R, 15R and 18R have been observed in Fe–Mn–Si-based shape memory alloys and Fe–Mn–C-based alloys [21–23]. They are regarded as transition phases or long-period SF structures of ε-martensite, and their formation is observed during the phase transformation of austenite → ε-martensite, which is different from the present case showing the formation of LPO structure from perfect ε-martensite.
Figure 5. (a) Engineering stress–engineering strain curve and (b) normalized work hardening rate as a function of true strain.

![Figure 5](image)

Figure 6. (a) Two types of SF in hcp structure [18] and (b) SFP of two types of SF in ε-martensite.

![Figure 6](image)

In fact, Bracke et al [24] reported that one of the ε-martensite plates contains small regions of austenite when two ε-martensite plates intersect forming α′-martensite. They claimed that the formation of α′-martensite causes internal stresses since it has a higher volume than the parent ε-martensite and, as a result, the dislocations at the interface become mobile and locally retransform the ε-martensite into austenite, thereby relaxing some of these internal strains. The same argument can be applied to explain the formation of LPO structure in the present case. As shown in figure 7, the formation of LPO structure has been found in the ε2-martensite plate impinging on the ε1-martensite plate which formed α′-martensite within the plate. In such a case, the strain accumulated in the ε1-martensite plate can be relieved by the formation of α′-martensite. On the other hand, in the case of the ε2-martensite plate, the generation of mobile dislocation at the interface between ε2-martensite and the newly formed α′-martensite can induce the deformation of ε2-martensite plate in a direction opposite to the applied stress, resulting in the formation of LPO structure within ε2-martensite plate and eventual occurrence of reverse transformation of ε-martensite into austenite. However, analysis of the EBSD results shown in figure 1 indicates that most of the reverse transformation occurs without involving the formation of α′-martensite. Schmid factors of all four possible variants of ε-martensite have been calculated as shown in figure 8. Four variants are marked by blue, purple, light blue and yellow colors and their respective Schmid factors are shown. The ε-martensite plates marked by red and green colors are from another parent austenite grain and annealing twins, respectively. It shows that the blue-colored ε-martensite plates having the largest Schmid factor among the four variants are seen to grow and nucleate more frequently than others. On the other hand, the yellow-colored ε-martensite plates susceptible to reverse transformation have the lowest Schmid factors of 0.113 among the four variants. Therefore, the deformation of these yellow-colored ε-martensite plates is quite restricted. It is quite possible that these yellow-colored ε-martensite plates are subjected to compressive stress due to the deformation of the surrounding austenite, resulting in their reverse transformation to austenite.

4.2. Effect of deformation-induced transformation on work hardening

As shown in figure 5(b), the normalized work hardening rate gradually increases with increasing deformation up to the strain of about 0.1, after which it gradually decreases. Comparison of the variation in the normalized work hardening rate with those in the phase fractions
of constituent phases with deformation (figure 4) shows that the deformation-induced phase transformation behavior mainly affects the work hardening behavior. As shown in figure 4, the deformation-induced phase transformation of austenite $\rightarrow \varepsilon$-martensite starts at the strain of 0.02, while that of $\varepsilon$-martensite $\rightarrow \alpha'$-martensite starts at the strain of 0.07. As compared to the initial phase fractions before deformation, at the strain of 0.12 where the steel shows the maximum normalized work hardening rate, the phase fraction of austenite decreases by 34% and those of $\varepsilon$-martensite and $\alpha'$-martensite increase by 24 and 10%, respectively, indicating that most of the deformation-induced phase transformation up to the strain of 12% is associated with austenite $\rightarrow \varepsilon$-martensite transformation. Moreover, the maximum normalized work hardening rate is obtained when the amount of $\varepsilon$-martensite is maximum, although there is a small difference in the amounts of strain showing two maxima due to the experimental setup. This result indicates that the deformation-induced phase transformation of austenite $\rightarrow \varepsilon$-martensite is more effective in work hardening than that of $\varepsilon$-martensite $\rightarrow \alpha'$-martensite. The increase in work hardening rate with an increase in the phase fraction of $\varepsilon$-martensite found in the present study is in agreement with the result of previous investigations showing that the steels show extremely steep work hardening behavior when the deformation-induced $\varepsilon$-martensite plate collides against a pre-existing $\varepsilon$-martensite plate with a different {111}$\gamma$ habit plane [25]. It has also been claimed that only a single type of partial dislocation is active in a given slip system for the formation of deformation-induced $\varepsilon$-martensite, resulting in a relatively large accommodation strain at the interface [6].

On the other hand, the deformation-induced phase transformation of $\varepsilon$-martensite $\rightarrow \alpha'$-martensite does not appear to contribute to an increase in work hardening rate of the present steel. It has been suggested [6] that the deformation-induced $\varepsilon$-martensite $\rightarrow \alpha'$-martensite transformation plays two contrasting roles in work hardening: $\alpha'$-martensite

**Figure 7.** (a) TEM micrograph of the deformed $\varepsilon_2$-martensite plate shown in figures 3(a) and (b) the SAD patterns corresponding to each region.

**Figure 8.** Four possible variants of $\varepsilon$-martensite and their respective Schmid factors.
formation is responsible for the initially lowered work hardening rate, as well as the increases during later stage deformation due to the accumulation of $\alpha'$-martensite laths, resulting in a stress–strain curve of anomalous shape. In the present case, the phase fraction of $\alpha'$-martensite after fracture is only 28%, which is much smaller than the 78% of the binary 17Mn steel, showing low and high work hardening rates at early and late stages of deformation, respectively [6]. The decrease in the work hardening rate associated with the $\varepsilon$-martensite $\rightarrow \alpha'$-martensite transformation is due to the so-called ‘window effect’, which reduces the high internal stresses generated locally by the blockage of plastic flow in austenite by pre-existing $\varepsilon$-martensite plates [26]. The consequence of a lowered work hardening rate due to deformation-induced phase transformation of $\varepsilon$-martensite $\rightarrow \alpha'$-martensite at the later stage of deformation is the reduction in ductility. The total elongation of the present Fe–17Mn–0.02C steel is 26%, which is much smaller than the about 40% of the binary Fe–17Mn steel [6]. It is believed that the inactive formation of $\alpha'$-martensite in the present steel is due to the increased mechanical stability of austenite and $\varepsilon$-martensite by C addition. Another factor contributing to the lowered work hardening rate after the strain of 10% is the reverse transformation of $\varepsilon$-martensite $\rightarrow$ austenite. Since $\varepsilon$-martensite plates are strong obstacles to plastic flow, their disappearance by reverse transformation results in the softening of the steel.

5. Conclusions

The deformation behavior of Fe–17Mn–0.02C consisting of austenite and $\varepsilon$-martensite has been investigated, with particular emphasis on the effect of deformation-induced phase transformation on work hardening and resultant tensile properties.

1. Deformation-induced phase transformation occurs via austenite $\rightarrow \varepsilon$-martensite $\rightarrow \alpha'$-martensite as well as the direct transformation of austenite $\rightarrow \alpha'$-martensite at high stress level.
2. The phase fraction of austenite continuously decreases with increasing deformation up to the strain of 0.12, but increases with further deformation, indicating the occurrence of reverse transformation of $\varepsilon$-martensite $\rightarrow$ austenite during deformation. The occurrence of reverse transformation has been confirmed by EBSD and in situ TEM analyses.
3. Reverse transformation of $\varepsilon$-martensite $\rightarrow$ austenite is linked to the formation of SFs in $\varepsilon$-martensite. TEM analysis of the $\varepsilon$-martensite containing SFs shows that it is a 6H LPO structure characterized by the presence of SFs in every sixth layer.
4. The comparison of the variation in the normalized work hardening rate with those in the phase fractions of constituent phases with deformation reveals that the transformation of austenite $\rightarrow \varepsilon$-martensite is more effective in work hardening than that of $\varepsilon$-martensite $\rightarrow \alpha'$-martensite.

Acknowledgments

We thank Dr Jae Suk Jeong and Professor Yang Mo Koo for carrying out the x-ray diffraction analysis and POSCO for financial support. Ki Hyuk Kwon and Byeong-Chan Suh have contributed equally to this work.

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