Micro-plasticity of medium Mn austenitic steel: Perfect dislocation plasticity and deformation twinning

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Abstract
The micro-scale plastic deformation behavior of an austenitic Fe-1.2%C-7.0%Mn (in wt%) steel was studied by means of nano-indentation and in situ compression of micro-pillars with selected crystallographic orientations. Transmission electron microscopy analysis reveals that the partial dislocation mediated twinning is preferred in a [001]-oriented single grain under compression. Twinning leads to large strain bursts during the nano-indentation and the micro-pillar compression. Perfect dislocation slip is the dominant deformation mechanism in a [111]-oriented single grain under compression. The observations offer strong support for the hypothesis that deformation twinning is a plasticity enhancing mechanism activated during the deformation of medium Mn steel.

1. Introduction

The strong global interest in medium Mn (4–7%) steel is due to their excellent strength-ductility balance resulting from their specific strain hardening behavior. Their properties are achieved despite much lower alloying additions as compared to high Mn twinning induced plasticity (TWIP) steel. A medium Mn steel typically achieves an ultimate tensile strength (UTS) more than 1 GPa and a total elongation (TE) in the range of 30–40% [1–3]. The excellent mechanical properties of medium Mn steel are associated with a two-phase microstructure consisting of austenite and ferrite.

The deformation mechanism of the austenite phase in high Mn TWIP steel has been the subject of numerous studies [4–6]. The beneficial influence of twins on the plasticity of FCC metals is well documented [7,8]. The stacking fault energy (SFE) of the austenite affects the deformation mechanism. Martensitic transformation, leading to transformation-induced plasticity (TRIP) effect, is the predominant deformation mode when the SFE is less than 15 mJ m⁻². The TWIP effect becomes dominant when the SFE is within the 15–45 mJ m⁻² range [9–11].

It has been suggested that both TRIP and TWIP effects are activated in the retained austenite of medium Mn steel [12–14]. The mechanical properties of the retained austenite in medium Mn steels have however not yet been documented. One reason for this is that the grain size of the retained austenite in intercritically annealed medium Mn steels is often very small, typically less than 1 μm. The ultra-fine grain size of the retained austenite makes it challenging to evaluate their intrinsic properties. Furthermore, the carbon and Mn contents of the retained austenite in the intercritically-annealed medium Mn steel are very different from the nominal steel composition, as carbon and Mn partition from ferrite to austenite during intercritical annealing.

The present work aims at revealing the orientation-dependent deformation mechanisms of a medium Mn austenitic steel. In the present study, a coarse-grained bulk sample of fully austenitic medium Mn steel was produced to study the micro-plasticity of the high carbon, medium Mn austenitic phase by means of both nano-indentation and in situ compression of micro-pillars with selected crystallographic orientations. Transmission electron microscopy (TEM) was used to investigate dislocation interactions and the evolution of the deformation microstructure after nano-indentation and micro-pillar compression tests.
2. Experimental procedures

The composition of the hot rolled steel used in the present study was Fe-1.2%C-7.0%Mn (in wt%). This corresponds to the typical composition of the austenite phase in intercritically annealed medium Mn steels, when the annealing is carried out to obtain the maximum volume fraction of austenite at room temperature. The microstructure of the hot-rolled sheet steel was made fully austenitic by water quenching from 1150 °C. The selection of the grains with specific orientations was determined by electron backscatter diffraction (EBSD) in an FEI Quanta 3D FEG scanning electron microscopy (SEM). The specimens used for the EBSD analysis were prepared by electro-chemical polishing in a solution of 5% HClO4 analysis were prepared by electro-chemical polishing in a solution of 95% CH3COOH. Coarse grains with the surface normal oriented close to the [001] and [111] directions were chosen for nano-indentation and micro-pillar compression tests.

Nano-indentation tests were conducted using a Hysitron PI 85 SEM Pico-Indenter equipped with a diamond Berkovich tip. The load was applied to a maximum depth of 40 nm. The nano-indentation tests were performed at a displacement rate of 8 nm/s at the initial loading stage. TEM samples were taken from the indentation-tested area by the FIB lift-out technique in an FEI Helios Nanolab 650 dual beam FIB. The Oliver and Pharr analysis method [15] was used to analyze the nano-indentation force-displacement data. The hardness (H) and the reduced modulus (Er) were derived from the following equations:

\[ H = \frac{P_{\text{max}}}{A} \]  
\[ \text{(1)} \]

and

\[ E_r = \frac{1}{\beta} \left( \frac{S}{2} \right) \frac{A}{\sqrt{A}} \]  
\[ \text{(2)} \]

where \( P_{\text{max}} \) is the maximum load applied during the indentation, \( A \) is the projected area of contact between the indenter and the specimen, \( \beta \) is a correction factor which depends on the geometry of the indenter (\( \beta = 1.034 \), for the Berkovich-type indenter), and \( S \) is the unloading stiffness at the maximum depth of penetration. Since elastic deformation occurs in both the specimen and the indenter, \( E_r \) is described by the following relationship

\[ E_r = \left( 1 - \frac{v_s^2}{E_s} \right) + \left( 1 - \frac{v_i^2}{E_i} \right) \]  
\[ \text{(3)} \]

\( E_s \) and \( v_s \) are Young’s modulus and Poisson’s ratio for the specimen. \( E_i \) and \( v_i \) are Young’s modulus and Poisson’s ratio for the indenter. For the diamond Berkovich indenter, \( E_i = 1141 \) GPa and \( v_i = 0.07 \).

In order to make accurate measurements by indentation experiments, the projected contact area, \( A \), must be precisely known. The most commonly used method to calibrate the shape of the indenter tip was proposed by Oliver and Pharr [15,16]. \( A \) is empirically derived as a function of contact depth (hc), i.e. \( A = f(h_c) \). The value of \( h_c \) is estimated from

\[ h_c = h_{\text{max}} - 0.75 \frac{P_{\text{max}}}{S} \]  
\[ \text{(4)} \]

where \( h_{\text{max}} \) is the maximum depth of penetration. The area function of \( f(h_c) \) was established by means of a tip calibration using a fused silica standard. The area function relating the projected contact area (A) to the contact depth (hc) was determined as

\[ A = 24.5 h_c^2 - 5.2935 \times 10^4 h_c + 4.7361 \times 10^6 h_c^{1/2} - 5.1554 \times 10^7 h_c^{1/4} + 1.3349 \times 10^8 h_c^{1/8} - 8.7237 \times 10^8 h_c^{1/16} \]  
\[ \text{(5)} \]

where the first term, 24.5\( h_c^2 \), describes an ideal Berkovich tip and the other terms describe the deviation from the ideal tip geometry.

The micro-pillars of approximately 600 nm diameter with a height-to-diameter aspect ratio of 3 were fabricated in an FEI Helios Nanolab 650 dual beam FIB operated at 30 kV. The taper angle of the micro-pillars was approximately 3°. In-situ compression tests on the micro-pillars were performed in a SEM using a Hysitron PI 85 SEM Pico-Indenter. A flat diamond punch tip with a diameter of 1 μm was used. The load was applied up to a maximum depth of 150 nm. The compression tests were performed at a strain rate of 0.02 s⁻¹. The morphology of the deformed micro-pillars was analyzed in a ZEISS field emission Ultra-55 SEM before and after the compression tests. TEM samples of the deformed pillars were prepared by the FIB lift-out technique. TEM observations were carried out in a FEI JEM-2100F FE-TEM operating at 200 keV.

3. Results

3.1. Nano-indentation tests

Fig. 1 shows a SEM micrograph and EBSD normal direction (ND) inverse pole figure (IPF) maps of the fully austenitic Fe-1.2%C-7.0%Mn steel. The average grain size was approximately 80 μm. Recrystallization twins were commonly observed. The twin boundaries are indicated by white lines in the IPF maps. Both nano-indentation and micro-pillar compression tests were performed in coarse austenite grains for which the surface normal was oriented close to the [001] and [111] directions (Fig. 1). The influence of grain orientation on the twinning behavior of FCC metals and alloys is well documented [17–20]. The stress on the leading and trailing partials of dissociated dislocations depends on the orientation of the compression axis and the difference in the stress affects the stacking fault width [17]. Deformation twinning is favored in [001]-oriented crystals when subjected to compression deformation, while perfect dislocation glide is preferred in [111]-oriented crystals in compression [17,20]. The calculated maximum Schmid factors for the perfect and partial dislocations with respect to the [001] and [111] compression axes are listed in Table 1.

Fig. 2 shows the crystallographic orientation dependence of the nano-indentation test results for the [001] and [111] directions. The nano-indentation test results of the present study and the literature data [20–24] are summarized in Table 2. A hardness of 7.6 GPa was obtained when the indentation load was applied along the [001] direction (Fig. 2(a)). Young’s modulus, \( E_{[001]} \), was estimated to be 234.5 GPa. When the indentation was applied along the [111] direction (Fig. 2(b)), the hardness and Young’s modulus, \( E_{[111]} \), were 8.5 GPa and 271.5 GPa, respectively. This difference, i.e. Young’s modulus in the <111> direction being higher than that in the <001> direction, is well-documented for austenitic alloys [20,25].

Due to the limitations of the microfabrication process, the indenter tip is not ideally sharp, but rounded. The geometry of the indenter tip can be approximated as spherical in the earliest stages of contact where plastic yielding occurs [22–24,26]. With this approximation, it is possible to predict the expected elastic response using the Hertzian law for mechanical contacts, as reported in previous studies [22–24,26]. The elastic portion of the load-displacement curve (P-h curve) can be described by the Hertzian elastic contact solution [27]:
\[ P = \frac{4}{3}E_r \sqrt{rh^3} \]  
\[ \tau_{\text{max}} = 0.30 \left( \frac{6P_r E_s^2}{\pi^3 r^2 E_r} \right)^{1/3} \]

\( P \) is the applied load, \( h \) is the corresponding indentation depth, and \( r \) is the radius of the indenter tip. The radius of the indenter tip used in the present study was determined to be 175 nm by the tip calibration method. The elastic portion of the load-displacement predicted by the Hertzian solution is indicated in Fig. 2(a) and (b).

In both nano-indentation force-displacement curves shown in Fig. 2, the first pop-in loads, \( P_c \), i.e. the load at which the first displacement burst occurs, were noticeably different. The \( P_c \) in the [001] direction was 49.0 mN at a displacement of 5.9 nm. The \( P_c \) in the [111] direction was much higher, 97.9 mN at a displacement of 8.5 nm. The maximum elastic shear stress underneath a spherical indenter, \( \tau_{\text{max}} \) can be calculated using the following relation [27]:

Table 1

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<tr>
<th>Compression axis</th>
<th>Schmid factors</th>
<th>Perfect dislocation</th>
<th>Partial dissociations</th>
<th>Twinning partials</th>
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<td>Leading</td>
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<td>8 systems</td>
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<td>4 systems</td>
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<tr>
<td>[111]</td>
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<td>Leading</td>
<td>0.16</td>
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<td></td>
<td>6 systems</td>
<td>0.31</td>
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<td>6 systems</td>
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Table 2

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<th>Alloy</th>
<th>Phase</th>
<th>Orientation</th>
<th>( E_s ) (GPa)</th>
<th>( E_r ) (GPa)</th>
<th>( H ) (GPa)</th>
<th>( \tau_{\text{max}} ) (GPa)</th>
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<td>[101]</td>
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<td>172.4</td>
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<td></td>
<td></td>
<td>[111]</td>
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<td>[001]</td>
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<td>247.7</td>
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<td>–</td>
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<td>Medium Mn Steel [Present Study]</td>
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Fig. 1. (a) SEM backscattered image of the austenitic Fe-1.2%C-7.0%Mn steel. (b) EBSD IPF map of the area labelled “b” in (a). (c) EBSD IPF map of the area labelled “c” in (a). The circles in (b) and (c) indicate the positions of [001]/ND-oriented and [111]/ND-oriented austenitic grains, respectively, where nano-indentation and micro-pillar compression tests were performed. Colors in the IPF maps indicates the crystal directions parallel to the viewing plane. The white lines in the IPF maps indicate the recrystallization twin boundaries.

Fig. 2. Nano-indentation force-displacement curve for (a) the [001]/ND-oriented austenitic grain and (b) the [111]/ND-oriented austenitic grain in the medium Mn austenitic steel.
note that when the indentation was tested along the [001] direction in which the twinning is expected to be favored, pop-ins were more frequently observed, as indicated by the arrows in Fig. 2(a). An indentation behavior with multiple pop-ins is clearly associated with deformation twinning.

In order to investigate the deformation mechanisms, TEM

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**Fig. 3.** Deformation-induced twinning in the medium Mn austenitic grain indentation-tested along the [001] direction. (a) Cross-sectional TEM micrograph of the deformed austenite grain, viewed along the [110] zone axis (inset: corresponding diffraction pattern showing the presence of reflections due to the twinned austenite). (b) DF image of the mechanical twins in the area indicated by the rectangle in (a). (c) HR lattice image of the mechanical twins (inset: corresponding Fast Fourier transform).

**Fig. 4.** Perfect dislocation slip in the medium Mn austenitic grain indentation-tested along the [111] direction. (a) Cross-sectional TEM micrograph of the deformed austenite grain, viewed along the (110) axis (inset: selected area diffraction pattern of the deformed austenite). (b) HR lattice image of dislocations on the (111) and (111) slip planes in the deformed austenite. TEM BF micrographs of the area of the deformed austenite, indicated by the rectangle in (a) for three different two-beam conditions: (c) $g = (002)$, (d) $g = (111)$, and (e) $g = (111)$. Note that the straight features in (b) and (c) are not associated with stacking faults. The contrast is due to the perfect dislocations gliding on (111) and (111) slip planes. The schematics in (c) to (d) illustrate the tetrahedron showing the orientation of (111) slip planes, and the perfect dislocations on (111) and (111) slip planes.
samples were prepared from the deformed region of the nano-indentation test by means of the FIB lift-out technique. Fig. 3 shows TEM micrographs of deformation-induced nano-sized twins in the [001]/ND-oriented austenite grain. The viewing direction was along the [110] zone axis. The TEM sample consisted of a single austenite grain of which the surface normal was oriented close to the [001] direction. The FIB milling during the TEM sample preparation did not induce phase transformation. The twin spots in the diffraction pattern [110] zone axis clearly indicate the presence of deformation twins. Streak-like patterns due to the nano-sized twins and the stacking faults were also visible in the diffraction pattern. Both the dark-field (DF) image and the high resolution (HR) lattice image show twins, with a thickness in the range of 4–10 nm. Although four equivalent twinning systems were in principle possible according to the Schmid factor analysis of Table 1, only one twin system, (T11)/C138, was activated.

Fig. 4 shows TEM micrographs of the [111]/ND-oriented austenite grain after the nano-indentation test. The viewing direction was along the [T10] zone axis. There was no indication for the formation of deformation twins in both the diffraction pattern (Fig. 4(a)) and the HR lattice image (Fig. 4(b)). Straight features, similar in appearance to stacking faults, were present along the [111] and (111) slip planes, as shown in the HR lattice image of Fig. 4(b) and the TEM two-beam bright field (BF) image shown in Fig. 4(c). In order to characterize those defects, TEM observations were carried out both near the [211] zone axis and the [2T1] zone axis. In these conditions, as shown in Fig. 4(d) and (e), only perfect dislocation contrast was visible, without any stacking fault contrast. This indicates that the defects formed on two different (111) slip planes were perfect dislocations. The straight features observed in the [T10] zone axis were therefore due to perfect dislocations on the (111) and (111) slip planes. The dislocation density was higher on the (110) plane as compared to the (111) plane (Fig. 4(b) and (c)). Slip on the (111) plane, which is perpendicular to the loading axis, should in principle not be activated. Some dislocations were however also observed on the (111) planes. This may be due to the fact that the loading axis was not precisely parallel to the [111] direction.

3.2. Micro-pillar compression tests

Fig. 5 shows the crystallographic orientation dependence of the micro-pillar compression results for the [001] and [111] directions. The micro-pillar test results are summarized in Table 3. There were distinct differences in the plastic deformation behaviors of the [001]- and [111]-oriented micro-pillars. Large-scale strain bursts followed by large stress drop were observed for the [001]-oriented micro-pillar, in which the twinning is expected to be favored (Fig. 5(a) and Supplementary Movie 1). The average strain burst size was 0.78 ± 0.56%. The accumulated burst strain was 4.7%. In contrast, the [111]-oriented micro-pillar, in which the dislocation glide is favored, exhibited frequent, small-scale strain bursts (Fig. 5(b) and Supplementary Movie 2). The average strain burst size and the accumulated burst strain were 0.29 ± 10% and 2.9%, respectively. The [111]-oriented micro-pillar had a smooth transition from elastic to plastic flow and a steady rate of work hardening, as compared to the [001]-oriented micro-pillar. The stress for yield onset in the [001]-oriented micro-pillar was slightly higher than in the [111]-oriented single crystal. However, the stress levels at the maximum strain of approximately 9% (i.e. a displacement of 150 nm) was similar for both cases, 2.2 GPa.

Supplementary video related to this article can be found at http://dx.doi.org/10.1016/j.actamat.2017.06.014.

The yield stress was clearly larger for the [001]-oriented micro-pillar (0.84 GPa) as compared to the [111]-oriented micro-pillar (0.64 GPa). Here, the yield stress was considered to be the stress at which the elastic-plastic transition is observed, as indicated by the arrow in Fig. 5(a) and (b). Using the Schmid factor, $m$, for each compression direction (Table 1), the critical resolved shear stress, $\tau_{crss}$, was calculated using the equation $\tau_{crss} = m \cdot \sigma_{ys}$ where the $\sigma_{ys}$ is...
the yield stress. The $\tau_{\text{crss}}$ for the [001] direction (0.40 GPa) was much higher than $\tau_{\text{crss}}$ for the [111] direction (0.17 GPa) (Table 3). Young’s modulus of the specimen, $E_s$, was estimated from the stiffness of the elastic portion of the compression stress-strain curves. The $E_s$ for the [001] direction (82 GPa) was lower than that for the [111] direction (102 GPa).

Fig. 6 shows a [001]-oriented austenite grain micro-pillar deformed in compression to a strain of 10%. Clear slip traces for the (111) slip plane were visible in the SEM image of the deformed crystal surface (Fig. 6(a)). These traces are due to the activation of the (111)\{TT2\} twin system. The primary twin system activated during the micro-pillar compression, (111)\{TT2\}, was different from the one activated during the nano-indentation, (T11)\{TT2\}, despite the fact that both tests were carried out in the same austenitic grain. In order to observe the deformation twins from the edge-on direction, the TEM specimen of the deformed micro-pillar was taken in a manner that the electron beam was aligned parallel to the [1T0] zone axis. Note that the TEM specimen taken after the nano-indentation was viewed along a different zone axis, the [110] zone axis (Fig. 3). Fig. 6(b) shows a TEM BF image of the deformed micro-pillar. Fig. 6(c) and (d) show TEM DF images of the nano-sized twins in the deformed micro-pillar. Three relatively thick twins of the (111)\{TT2\} twin system were observed (Fig. 6(c)). A single (11T)\{112\} twin was also activated (Fig. 6(d)). The thickness of the twins was in the range of 10–32 nm. A low density of stacking faults and dislocations was also observed in the upper part of the deformed micro-pillar. It is important to note that the FIB milling during the micro-pillar preparation induced the austenite-to-ferrite transformation at the surface of the micro-pillar. Fig. 6(e) shows that the deformed micro-pillar was covered by a layer of ferrite. The average thicknesses of the surface transformed to ferrite was 34.6 ± 6.5 nm. It is clear that this ferrite layer was formed during the FIB micro-pillar fabrication and not during the micro-pillar compression because no ferrite layer was observed in the TEM samples of the austenite grains after nano-indentation. The inset in Fig. 6(e) shows a selected area diffraction pattern indicating the Kurdjumov-Sachs (K-S) orientation relationship between the austenitic matrix and the surface ferrite layer. The origin of the surface transformation to ferrite will be discussed in section 4.4.

Fig. 7 shows a [111]-oriented austenite grain micro-pillar deformed to 10% engineering strain in compression. Slip traces parallel to the (TT1) slip plane are visible at the deformed crystal surface (Fig. 6(a)). A very high density of dislocations with the primary slip system of the (TT1) planes was observed in the deformed micro-pillar (Fig. 7(b)). Similarly to the observations made for the TEM sample of the indentation-tested, [111]-oriented grain (Fig. 4), straight features corresponding to the perfect dislocations on the (TT1) slip planes were frequently observed (Fig. 7(c)). Their Burgers vector was either a/2[101] or a/2[011]. These perfect dislocations were out of contrast for the $g$ = [TT1] two-beam condition (Fig. 7(d)), indicating that the product of their Burgers vector and the g vector was zero, i.e. [101]·(TT1) = 0 and [011]·(TT1) = 0. Together with the activation of the primary slip on the (111) slip plane, a high density of tangled dislocations, which do not appear straight when viewed along the [1T0] zone axis, was also observed. Fig. 7(e) and (f) show that the FIB-induced austenite to ferrite transformation was also observed in the [111]-oriented micro-pillar. The deformed micro-pillar was surrounded by a layer of the transformed ferrite with a thickness of 22.3 ± 2.3 nm. The inset in Fig. 7(e) shows a selected area diffraction pattern indicating the Nishiyama-Wasserman (N-W) orientation relationship between the austenitic parent matrix and the transformed ferrite surface.

4. Discussion

4.1. Orientation dependent deformation behavior

The motion of isolated Shockley partial dislocations with the same a/6<112>-type Burgers vector on successive [111]-type planes is the origin of overlapping stacking faults and deformation-induced twins. The SFE plays an important role in deformation twinning. The room temperature SFE of the Fe-1.2%C-7.0%Mn used...
in the present study is estimated to be 34 mJ m$^{-2}$, 28 mJ m$^{-2}$ and 28 mJ m$^{-2}$ based on the thermodynamic models proposed by Curtze and Kuokkala [6], Saed-Akbari et al. [10], and Dumay et al. [28], respectively. An $\gamma/\varepsilon$ interface energy of 10 mJ m$^{-2}$ was used for the calculation of the SFE. As reported in Refs. [9,11], the TWIP effect is the predominant plasticity-enhancing deformation mode when the SFE is in the range of 15–45 mJ m$^{-2}$. The previous reports on deformation behavior of austenitic steels [9,11] are consistent with the observations of the present study that twinning or dislocation gliding is activated during the deformation of the medium Mn austenitic steel and that no austenite to $\gamma'$- or $\varepsilon$-martensite transformation is observed. It should be pointed out that the models developed for the SFE calculation were derived for steels with a relatively high Mn content ranging from 10 to 35 wt% and that precise experimental values of the SFE for medium Mn austenitic steels are unknown.

It was found that the deformation mode during the nanoindentation and the micro-pillar compression results was strongly influenced by the grain orientation. Deformation twinning was the preferred deformation mode in the [001]/ND-oriented single grain under compression. Perfect dislocation slip was the dominant deformation mechanism in the [111]/ND-oriented single grain deformed in compression. These observations are consistent with the results of previous studies [17,20]. In particular, Karaman et al. [17] studied the stress-strain behavior of the bulk sample of high Mn Hadfield steel (Fe-12.34Mn-1.03C in wt%) single crystals for selected crystallographic orientations ([TT1], [001] and [T23]) under tension and compression. They reported that twinning was the dominant deformation mechanism in [0 0 1] crystals subjected to compression, while multiple slip was determined to be the dominant deformation mechanism in [T11] crystals in compression.

Similar observations have been reported for the small-scale compression tests. Kang et al. [20] reported an evidence of deformation twinning in a high Mn TWIP steel (Fe-0.58C-17.5Mn-1.5Al in wt%) when the nano-indentation was applied along the [001] direction of the crystal. They reported that no mechanical twin was observed in the grain indentation-tested along the [111] orientation.

Choi et al. [29] reported similar trends of the orientation-dependent deformation behavior in high Mn TWIP steel (Fe-0.6C-22Mn in wt%) micro-pillars. The orientation dependent deformation behavior observed in the present study can be explained by a Schmid factor analysis, as shown by Karaman et al. [17]. Table 1 lists the calculated Schmid factors for slip and deformation twinning as a function of the compressive loading directions. When the compressive loading is along the [001] direction, the Schmid factor for the leading partial dislocation ($m_{tp}$) is larger than that for the trailing partial dislocation ($m_{slip}$). This effectivelly promotes the creation of wide stacking faults and twin formation. The $m_{tp}$ is also larger than the Schmid factor for the perfect dislocation slip ($m_{slip}$). In contrast, when the loading axis was parallel to the [111] direction, the twin formation is suppressed since $m_{tp} < m_{slip}$. If it is assumed that the critical resolved shear stresses, $\tau_{crss}$, for both twinning and slip are approximately equal, further prediction of deformation behavior is possible, as shown by Gutierrez-Urrutia et al. [18]. In compression along the [001] direction, deformation twinning dominates because the Schmid factor for the twin system is higher than that for slip, i.e., $m_{tp} > m_{slip}$. In compression along the [111] direction, the perfect dislocation slip should be dominated as $m_{tp} < m_{slip}$. However, in principle, $\tau_{crss}$ for twinning and slip are not equal and thus a careful analysis is needed. $\tau_{crss}$ can be derived by several approaches, as will be discussed in section 4.2.

In general, the activated slip/twin systems are those predicted by Schmid’s law. If the compressive axis is precisely along the [111] direction, there are six slip systems with an equivalent Schmid factor (Table 1). However, a slight misorientation of the compressive axis from the exact [111] direction causes only one specific slip system to be activated. Table 4 lists the calculated maximum Schmid factor for slip/twin systems considering the actual crystal orientation determined by EBSD and the experimentally observed twin/slip systems. When the compressive axis was along the [28 32 27] direction, the highest Schmid factor was found for the twin slip system. This slip system corresponds to the experimentally observed slip system. When the compressive axis was

![Fig. 7. SEM micrograph and cross-sectional TEM micrographs of medium Mn austenitic steel micro-pillars compressed along the [111] direction. (a) SEM image of the deformed micro-pillar viewed at an angle of 52° to the normal of the pillar. The tetrahedron in (a) indicates the orientation of the primary (TT1) slip plane. The deformation is by the motion of perfect dislocations with Burgers vector a/2[101] or a/2[011]. (b) TEM BF image of the deformed micro-pillar containing a high density of perfect dislocations. (c) Two-beam (g = (TT1)) TEM BF micrographs of the area indicated by the rectangle in (b), showing perfect dislocations on the (TT1) slip plane. (d) Two-beam (g = (TT1)) TEM BF micrographs of the same region. The perfect dislocations on (TT1) slip plane are out of contrast for g = (TT1). (e) TEM DF image taken using the (002) reflection, showing the ferrite layer formed at the surface of the micro-pillar during the FIB micro-pillar preparation (inset: corresponding diffraction pattern showing the N-W orientation relationship between the parent austenite (\(\gamma\)) and the transformed ferrite (\(\alpha\)). (f) TEM DF image taken using the (002), reflection, showing the austenite matrix in the deformed micro-pillar.](image-url)
The multiple pop-ins recorded during the nano-indentation in the [001] direction (Fig. 2(a)) were interpreted as being due to deformation twinning. In the micro-pillar compression tests, the orientation-dependence of mechanical behavior was more clearly shown. The large strain bursts during the compression of the [001]-oriented micro-pillar were clearly due to the rapid twin growth. These strain bursts plasticity are different from those appearing in micro-pillar associated with the continuous operation of a few dislocation sources [31–33], or the destruction of jammed dislocation configurations [34,35]. Similar strain bursts due to the twin growth in micro-pillars of a high Mn TWIP steel [36,37] and a single-crystal Ti alloy (HCP) have been reported. The percentage of the plastic strain contributed by deformation twinning, \( \varepsilon_t \), was estimated by means of the following equation [36]:

\[
\varepsilon_t = \frac{T \cdot \gamma \cdot \cos \alpha}{H}
\]

(8)

where \( T \) is the twin thickness, \( \gamma \) is the twinning shear strain equal to \( \sqrt{2}/2 \), \( \alpha \) is the angle between the pillar axis and the activated twinning direction, and \( H \) is the height of the pillar before the compression. The value of \( \varepsilon_t \) was 3.3%, which is smaller than the accumulated burst strain of 4.7% (Table 3). This difference may be due to the exclusion of the plasticity contributed by the dislocation glide and the formation of stacking faults. When the loading axis was along the [111] direction, i.e. when a slip-dominant deformation was favored, the accumulated burst strain was 2.9%. This was much smaller as compared to [001] direction, despite the fact that greater number of strain bursts took place during the micro-pillar compression. The observations offer a strong support for the hypothesis that deformation twinning is activated as a plasticity enhancing mechanism during the deformation of medium Mn steels.

Wu et al. [39] studied the mechanism of deformation twinning in a high Mn TWIP steel (Fe-0.6C-22.0Mn in wt%) micro-pillar. They reported that twin nucleation and growth occur via the emission of partial dislocations from the pillar surface on consecutive [111] planes. Their result is consistent with the results of the present study (Fig. 6) that the twin nucleation in micro-pillars invariably occurred near the pillar surfaces. The twin nucleation is possibly aided by the defects created during the ion-milling step. On this basis, Liang et al. [37] have proposed a mechanism for the formation of twins in a high Mn TWIP steel micro-pillar. The mechanism proposed by Liang et al. [37] involves the dissociation of radiation-induced a/3<111> vacancy Frank loops into sessile a/6<110> dislocations and twin-emitting a/6<112> Shockley partials on a conjugate (111) plane.

In the present study, the thickness of the deformation twins in the deformed micro-pillar ranged from 10 to 32 nm (Fig. 6). Considering the planar spacing of (111) plane in austenite, the glide of a/6<112>-type twinning partial dislocations on the approximately 50–150 successive (111) planes is required to form the observed deformation twins. Twin growth can occur in an avalanche-like manner, e.g. via the rapid and coherent partial dislocation emission adjacent to twin boundary as reported by Liang and Huang [36]. Liang and Huang observed a large strain burst due to twin growth in a high Mn TWIP steel (Fe-0.6C-22.0Mn in wt%) micro-pillar, similar to the one observed in the present study (Fig. 5(a)). They reported that the average rate of partial dislocation emission was as high as \( \sim 3300 \text{s}^{-1} \). Yu et al. [38] also observed a similar burst-like twinning behavior in a Ti alloy (HCP) micro-pillar. They described deformation twinning as a perfectly coherent “stimulated slip” phenomenon, which is in contrast to the less coherent “spontaneous slip” of ordinary dislocation plasticity. Their view was based on the fact that deformation twinning is characterized by perfectly correlated layer-by-layer shearing because all twinning partial dislocations on the adjacent parallel planes must have the same Burgers vector. This may explain why the deformation twinning causes large strain bursts. On the other hand, inelastic shear activities are randomly dispersed among slip planes in perfect dislocation slip process [38].

### Table 4

<table>
<thead>
<tr>
<th>Compression Axis</th>
<th>Actual axis</th>
<th>Maximum Schmid factors and the slip/twin system</th>
<th>Observed primary slip/twin system</th>
</tr>
</thead>
</table>
| [001]            | [1 1 50]    | \( \begin{array}{l}
\frac{0.416}{(T11)(T01)} \quad \frac{(111)(T02)}{0.480} \\
\frac{0.313}{(T11)(001)} \quad \frac{(T11)(T21)}{0.193}
\end{array} \) | \((T11)(T02)\) twin \( (111)(T02)\) twin |
| [111]            | [28 32 27]  | \( \begin{array}{l}
\frac{0.416}{(T11)(T01)} \quad \frac{(111)(T02)}{0.480} \\
\frac{0.313}{(T11)(001)} \quad \frac{(T11)(T21)}{0.193}
\end{array} \) | \((111)(T02)\) twin \( (111)(T21)\) twin |

4.2. Critical resolved shear stress for twinning and dislocation slip

The preference for twinning or slip can be explained by comparing the critical resolved shear stress for a perfect dislocation, \( \tau_{\text{perfect}} \), and the critical resolved shear stress to initiate the Shockley partial twinning dislocation \( \tau_{\text{partial}} \) to create a deformation twin. With an approximation of the source size equal to the grain size, \( \tau_{\text{perfect}} \) required to activate the dislocation source is given by classical dislocation theory [40],

\[
\tau_{\text{perfect}} = \frac{2aGb_{\text{perfect}}}{D}
\]

(9)

where \( G \) is shear modulus, \( b_{\text{perfect}} \) is the magnitude of the Burgers vectors of the perfect distortions, \( D \) is grain size, and \( \alpha \) is a constant describing the character of the dislocation (\( \alpha = 0.5 \) and 1.5 for edge and screw dislocations, respectively). \( \tau_{\text{partial}} \) can be obtained by an equation similar to Eq. (9), which takes into account the formation of the stacking fault. The stress for the twin growth from existing twin nuclei is determined by a balance between the repulsive forces of two Shockley partials and the attractive force of the SFE and it is given by \( \gamma/b_{\text{partial}} \). This leads to the following equation for \( \tau_{\text{partial}} \) [40]:

\[
\tau_{\text{partial}} = \frac{2aGb_{\text{partial}}}{D} + \frac{\gamma}{b_{\text{partial}}}
\]

(10)
where \( \gamma \) is the SFE, and \( b_{\text{partial}} \) is the magnitude of the Burgers vector of the partial dislocation. Equations similar to Eq. (10) have been proposed [18,41,42]. Application of Eq. (9) to the Fe-1.2%C-7.0%Mn steel used in the present study yields \( \tau_{\text{perfect}} = 69 \text{MPa} \) (\( G = 81 \text{GPa}, b_{\text{perfect}} = 0.256 \text{nm}, a \approx 1, \) and \( D = 0.6 \mu \text{m} \)). Eq. (10) yields \( \tau_{\text{partial}} = 229 \text{MPa} \) (\( \gamma = 28 \text{mJ m}^{-2}, b_{\text{partial}} = 0.148 \text{nm} \)). In both cases, the grain size was taken equal to the micro-pillar diameter. These estimates reveal that \( \tau_{\text{partial}} \) for deformation twinning should be considerably larger than \( \tau_{\text{perfect}} \) for the perfect dislocation glide. This trend is in agreement with the results of the present study that the critical resolved shear stress, \( \tau_{\text{crss}} \), for the \([001]\)-oriented micro-pillar, which is equivalent to \( \tau_{\text{partial}} \), was more than \( 2 \times \tau_{\text{perfect}} \) for the \([111]\)-oriented micro-pillar (Table 3).

The homogeneous dislocation nucleation model was initially proposed by Hirth and Lothe [43], and extended by Michalske and Houston [44]. In the model, the energies for the activation of a perfect dislocation Frank-Read source (\( E_{\text{perfect}} \)) and a partial dislocation twin source (\( E_{\text{partial}} \)) in the presence of the applied shear stress, are expressed as [43,44]:

\[
E_{\text{perfect}} = -\pi R^2 r_b \gamma + \frac{1}{2} G b_{\text{perfect}}^2 R \left( \frac{2}{1 - \nu} \left( \ln \frac{4R}{r_b} - 2 \right) \right) \tag{11}
\]

and

\[
E_{\text{partial}} = -\pi R^2 r_b \gamma + \frac{1}{2} G b_{\text{partial}}^2 R \left( \frac{2}{1 - \nu} \left( \ln \frac{4R}{r_b} - 2 \right) \right) + \pi R^2 \gamma \tag{12}
\]

where, \( R \) is a dislocation loop radius, \( r_b \) is the applied shear stress, \( \nu \) is the Poisson’s ratio, and \( r_b \) is the core radius of the dislocation, which is assumed to be equal to the Burgers vector. The maximum energy barrier occurs at a critical dislocation loop size corresponding to the condition, \( \partial E/\partial R|_{R=R_0} = 0 \). With this condition, the critical dislocation loop size and the maximum energy barrier can be calculated for a given applied shear stress. Fig. 8(a) and (b) show the applied shear stress dependence of the critical dislocation loop size and critical nucleation energy. The \( \tau_{\text{crss}} \) measured for the micro-pillars in this work is indicated in Fig. 8(a) and (b). It is shown that, as the applied shear stress increases, the critical loop size and the nucleation energy of partial and perfect dislocation loops become comparable. Fig. 8(b) shows that the creation of the partial dislocation loop is energetically favorable when the applied shear stress \( > 270 \text{MPa} \). When the applied shear stress is equal to the \( \tau_{\text{crss}} \) for the \([001]\)-oriented micro-pillar (400 MPa), \( E_{\text{partial}} \) is lower than \( E_{\text{perfect}} \), as indicated in Fig. 8(b). In this condition, the nucleation of partial dislocation loop is energetically favorable. When the applied shear stress equals the \( \tau_{\text{crss}} \) for the \([111]\)-oriented micro-pillar (170 MPa), \( E_{\text{perfect}} \) is significantly lower than \( E_{\text{partial}} \) (Fig. 8(b)), implying that the perfect dislocation glide is favorable. In other words, the preference for twinning in a \([001]\)-oriented micro-pillar and the preference for perfect dislocation glide in a \([111]\)-oriented micro-pillar are explained in terms of the energy of the dislocation nucleation.

In another approach to estimate the twinning stress, an empirical Hall-Petch-type relation has been used to describe the grain-size dependence of the twinning stress [45]:

\[
\sigma = \sigma_0 + \frac{K_T}{\sqrt{D}} \tag{13}
\]

where \( \sigma_0 \) is the stress at which yielding occurs at very large grain sizes, and \( K_T \) is the Hall-Petch slope for twinning. \( K_T \) has been reported to be consistently greater than \( K_s \), the Hall-Petch slope for deformation controlled by slip [45]. This implies that the grain size impact on twinning is more than its impact on slip. In FCC Cu, \( K_T \) is two times \( K_s \) [46]. Similar observations have been made for BCC [47–49] and HCP alloys [50]. A similar trend applies to micro-pillars. Yu et al. [38] studied the size-dependent deformation twinning behavior in HCP Ti alloy micro-pillars. They reported that the deformation twinning was entirely replaced by dislocation plasticity when the micro-pillar diameter was less than one micron. They explained that this is due to the fact that the stress required for deformation twinning increases drastically with decreasing sample size. If this argument is applied to the results of the present study, in the case of the micro-pillar compression test, \( \tau_{\text{partial}} \) is likely to be much larger than \( \tau_{\text{perfect}} \) because the stress required for deformation twinning is drastically increased in a small sample.

It should be mentioned that Choi et al. [29] reported the results in contradiction with the present results. They reported that the values of the \( \tau_{\text{crss}} \) of micro-pillars oriented for deformation twinning and micro-pillars oriented for dislocation glide were similar. They suggested that the elasto-plastic transition observed for the TWIP steel micro-pillars was due to dislocation glide, independent of the fact that the orientation may favor twinning. The reason for the apparent contradiction is not clear, but the discrepancy may have originated partly from difference in the SFE. The SFE of the Twip steel used in the work of Choi et al. [29] (23 mJ m\(^{-2}\)) was smaller than that of the steel used in the present work (28 mJ m\(^{-2}\)). Choi et al. [29] observed \( \varepsilon \)-martensite in the deformed TWIP steel micro-pillar, suggesting the SFE was clearly lower as compared to the steel used for the present work. If the SFE is smaller, the stress required for the twin growth decreases, leading to a decrease of \( \tau_{\text{perf}} \) according to Eq. (10).

The value for the \( \tau_{\text{crss}} \) obtained by the micro-pillar compression tests (\( \lesssim 0.40 \text{GPa} \)) was considerably lower than the value for the \( \tau_{\text{max}} \) obtained by nano-indentation (\( \sim 10 \text{GPa} \) \( = G/8 \)). Note that \( \tau_{\text{crss}} \) and \( \tau_{\text{max}} \) obtained in the present study are not physically same. \( \tau_{\text{crss}} \) is the maximum shear stress, which lies along the loading direction, i.e. it is not that resolved in specific slip direction. Bei et al. [51] also reported that the \( \tau_{\text{crss}} \) of a Mo alloy single crystal obtained from micro pillar tests (\( \approx G/26 \)) was a factor of 3 lower than \( \tau_{\text{max}} \) obtained from the nano-indentation tests (\( \approx G/8 \)). They argued that this difference was due to the different stresses required to homogeneously nucleate a full dislocation loop within a bulk sample during nano-indentation and to heterogeneously nucleate half or quarter dislocation loops at the free surfaces and edges of micro-pillars. Liang et al. [37] suggested that the ion-radiation-induced defects due to the FIB micro-pillar fabrication cause a local volume change in the damaged subsurface layer and induces substantial internal stress into this layer due to the constraint from those parts of the micro-pillar which are not affected by the ion-beam damage. They argue that the ion-radiation-induced internal stresses present in the damaged subsurface layer of micro-pillars facilitate the dissociation of vacancy Frank loops which act as twinning nuclei. In the present case, the phase transformation at the surface of the pillar created both surface internal stresses, due to the lattice expansion associated with the austenite-to-ferrite transformation, and a high density of the transformation dislocations.

### 4.3. Young’s modulus obtained by nano-indentation and micro-pillar test

The measured values of Young’s modulus were significantly different for nano-indentation and micro-pillar compression. For example, \( E \) for the \([001]\) direction was measured to be 234.5 GPa and 82 GPa by nano-indentation and micro-pillar compression, respectively. Pharr [16] reported that when the pile-up which forms around the indentation impression is prevalent in the
material, the Oliver and Pharr method for the nano-indentation data analysis may lead to an overestimation of the modulus by as much as 50%. He reported that pile-ups in the material will make a significant contribution when $h_{\gamma}/h_{\max} > 0.7$. Here, $h_{\gamma}$ is the final displacement after complete unloading and $h_{\max}$ is the maximum depth of penetration. In the present work, the value of $E_0$ obtained by the nano-indentation may have been overestimated as $h_{\gamma}/h_{\max}$ was approximately 0.71.

On the other hand, the values of Young’s modulus obtained by micro-pillar compression were considerably lower than the value of 159 GPa reported for an austenitic Fe-1.5 wt% C [52]. Young’s modulus is 174.7 GPa in the bulk sample of high Mn TWIP steel [53]. The underestimation of Young’s modulus in micro-pillar compression has been reported [54-56]. Greer et al. [55] reported that this anomalously soft initial response may partially be due to the compliance of the substrate supporting the micro-pillar. Choi et al. [57] reported that the underestimation of Young’s modulus is due to the substrate deformation and that this effect decreases with increasing values of aspect ratio. They provided the following equation, derived based on the Sneddon correction [58]:

$$\frac{1}{E_{\text{adj}}} = \frac{1}{E} \left(\frac{E^*}{E} + \frac{1}{4} \frac{d}{L_0}\right)$$  \hspace{1cm} (14)

where $E_{\text{adj}}$ is the adjusted Young’s modulus, $E$ is the apparent modulus measured by the micro-pillar test, $E^*$ is the reduced (indentation) modulus, $d$ is micro-pillar diameter, and $L_0$ is the initial micro-pillar height. The values of the $E_{\text{adj}}$ are listed in Table 3. These values are still too small. It is possible, as noted by Dimiduk et al. [56], that the misalignment between the loading plates and the specimen leads to the gradual elastic loading of the specimen and anomalous soft initial response of the specimen.

4.4. FIB-induced FCC-to-BCC transformation

Knipling et al. [59] reported that FIB milling resulted in the austenite-to-ferrite transformation in commercial stainless steels. Basa et al. [60] reported a very similar FIB-induced phase transformation in the highly stable austenite phase of a super duplex stainless steel. They concluded that the product BCC phase was not formed by a martensitic transformation based on the observation that the invariant plane strain and plastic deformation, which are expected in the case of a martensitic transformation, were not observed. They argued that the austenite-to-ferrite transformation was chemically-induced, i.e. it was due to the local enrichment of gallium, which is a strong ferrite stabilizer. Babu et al. [61] also observed the FIB-induced austenite-to-ferrite transformation in 316L austenitic stainless steel. They concluded that in addition to the local gallium enrichment, the strain associated with the ion implantation also promoted the phase transformation during the FIB irradiation. The FIB-induced austenite-to-ferrite transformation was also observed in the present work. A BCC ferrite layer with a thickness in the range of 20 nm—30 nm was formed at the surface of the micro-pillars during the FIB pillar microfabrication (Figs. 6(e) and 7(e-f)). Selected area diffraction patterns indicated both the K-S and N-W orientation relationship between austenite and ferrite. The product BCC layer is very likely not due to a martensitic transformation as the characteristic features of high carbon martensite were not observed by TEM analysis. This indicates that this transformation was mainly chemically-driven, as suggested by Basa et al. [60]. The mechanism of the phase transformation during the FIB microfabrication is different depending on the austenite stability. In the case of the highly stable austenite in the medium Mn steel used in the present study, the transformation was mainly chemically-driven. That is, during the gallium ion implantation, the composition of austenite was gradually changed and the austenite became metastable. Once a critical Ga content was reached, the austenite spontaneously transformed to ferrite. On the other hand, if the austenite is metastable, the phase transformation can be triggered mainly by the stress generated by the ion implantation. For example, the mechanically-induced martensitic transformation was observed during the FIB milling of the metastable retained austenite phase in a quenching and partitioning (Q&P) processed steel (Supplementary materials). Invariant plane-strain surface reliefs associated with the martensitic transformation were clearly observed in the retained austenite phase immediately after a single scan of FIB (Fig. 51). The structure of the BCC product phase was clearly identified by the EBSD analysis. Since this transformation took place after a single FIB scan with dwell time of microsecond time scale, the FIB-induced heating was negligible, i.e. phase transformation by diffusional mechanism should not be considered. As the contents of carbon and Mn, austenite stabilizers, were relatively low in the Q&P processed steel (Fe-0.4C-4.0Mn-1.6Si-1.0Cr in wt%), the stability of the retained austenite in this steel was clearly much lower as compared to the austenite in the medium Mn steel (Fe-1.2C-7.0Mn in wt%) used in the present study. The present results therefore suggest that the FIB-induced phase transformation should be seen as chemically-induced austenite-to-ferrite phase transformation when the austenite is highly stable.
For an austenite phase with low stability, martensitic transformation due to the strain induced by the ion implantation will take place.

A large density of dislocations is created during the austenite-to-martensite (γ → ℓ) transformation. Three families of dislocations are involved: (a) a/2<110>₁y-type transformation dislocations associated with the structural ledges, (b) a/2<111>ᵲ-type screw misfit dislocations at the austenite-martensite interphase boundary, and (c) a/2<110>₁y-type dislocations in austenite, resulting from the deformation of the austenite during the martensite transformation. The dislocations in the austenite are subsequently inherited by the martensite. The dislocations in lath martensite are mainly screw dislocations [62–65]. Shibata et al. [66] observed two sets of [112]₁ₓ<011>₂ₚ. Moritani et al. [63] compared the interphase boundary structures of the lath martensite and the bainitic ferrite lath. They observed similar accommodation screw dislocations in both cases. This implies that, regardless of whether the transformed layer observed at the surface of the micro-pillar is ferrite or martensite, the transformation creates a large density of screw-type dislocation segments. The micro-pillar tests are therefore influenced by the preexisting dislocations at the surface.

5. Conclusions

The micro-scale plastic deformation behavior of austenitic Fe-1.2C-7.0%Mn steel was studied in the [001] and [111] crystallographic orientations by means of nano-indentation and in situ micro-pillar compression tests.

1. The SFE of the steel was estimated to be in the range of 28–34 mJ m⁻². The calculated SFE range is consistent with the observations that, depending on the orientation, only twinning or dislocation gliding was activated during the deformation of the medium Mn austenitic steel.

2. Deformation twinning was the preferred deformation mode in the [001]//ND-oriented single grain under compression. Perfect dislocation slip was the dominant deformation mechanism in the [111]//ND-oriented single grain deformed in compression. The activated slip/twin systems were, in general, consistent with those predicted by the Schmid’s law. The rapid twin growth led to large strain bursts during the compression of the [001]//ND-oriented micro-pillar. The observations offer strong support for the hypothesis that deformation twinning is a plasticity enhancing mechanism activated during the deformation of medium Mn steel.

3. The critical resolved shear stress for [001]//ND-oriented micro-pillar, τₚₚₚₚₗₚₚₜₐₜₐ, was almost two times that for [111]//ND-oriented micro-pillar, τₚₚₚₚₗₚₚₚₜₚₑₚ₋ₑₚ. These results are in agreement with the calculation of the critical resolved shear stress based on the classical dislocation theory.

4. The FIB milling during the micro-pillar preparation induced the austenite-to-ferrite transformation at the surface of the micro-pillar. It is suggested that the mechanism of the FIB-induced phase transformation is different depending on the austenite stability. When the austenite is highly stable as the case for high carbon medium Mn steel (Fe-1.2C-7.0%Mn), the FIB-induced phase transformation was mainly chemically-induced due to the local enrichment of gallium, which is a strong ferrite stabilizer. For an austenite phase with low stability, as the case for the austenite in Q&P processed steel, martensitic transformation due to the strain induced by the ion implantation took place.

Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.actamat.2017.06.014.

References
