Effect of annealing treatment on the microstructure and mechanical properties of Fe-18Mn-0.8C-0.2 V TWIP steel

Jingshan Zeng¹, Chaoyue Chen²,³, ⁴, Jiang Wang¹, Zhongming Ren¹ and Wen Shi²,⁴

¹ Shanghai Aircraft Manufacturing Co. Ltd, Shanghai 201324, People’s Republic of China
² Sino-European School of Technology of Shanghai University, Shanghai 200444, People’s Republic of China
³ State Key Laboratory of Advanced Special Steels, School of Materials Science and Engineering, Shanghai University, Shanghai 200444, People’s Republic of China
⁴ Authors to whom any correspondence should be addressed.

E-mail: cchen1@shu.edu.cn and shiwen@staff.shu.edu.cn

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Abstract
The microstructure and mechanical properties of TWIP steels under different annealing treatments were investigated by static tensile tests, OM, TEM, and EBSD. Results show that the TWIP steel was dominated by full austenitic grains before and after tensile deformation. The TWIP steel exhibited optimal mechanical performance under the annealing treatment at 1000 °C, with the highest initial strain hardening rate \( \frac{d\sigma}{d\varepsilon} \). The grain size increased with the increasing annealing temperature, and the relationship between strain hardening and true strain turns into three stages from two stages. The strain hardening index \( n \) has the same transition trend under different annealing temperatures when \( \varepsilon < 0.07 \). With the increase of true strain, the \( n \) reached a maximum value of 0.53, 0.62, 0.70 at the annealing temperatures of 800 °C, 900 °C, 1000 °C, respectively. With the increase of the annealing temperature, the elements of V or Ti compounds exhibited a decrease in content but an increase in size. The fracture of the TWIP steel exhibited typical equiaxed dimples. Deformation twins with different orientations were generated within the austenitic grains after the deformation process. With the increase of annealing temperature, the decreased space between twins results in the strengthening of the TWIP effect.

1. Introduction
TWIP (twinning-induced plasticity) steels have received high interest in recent years due to their outstanding mechanical properties at room temperature [1–3]. The simultaneous high strength (ultimate tensile strength of up to 1100 MPa) and ductility (elongation to failure up to 100%) can be obtained based on a high work-hardening capacity [4–6]. Among the various TWIP steels, the high-Mn steel steels with an austenitic structure have captured increasing attention from the industry due to its excellent combination of mechanical properties. For example, TWIP steel has been widely used as the raw material for automotive steel sheets because of its excellent combination of weight reduction and improvement of mechanical property [1, 2]. As known to all, the superior performance of TWIP steel requires the microstructure with a single austenitic phase before plastic deformation, a number of annealing twins with specific sizes, and the formation of deformation twins (namely the TWIP effect). As for the stacking fault energy (SFE) above 18 mJ m\(^{-2}\), Allain [6] believed that it is prone for deformation twins to delay sample necking so that to enhance the elongation. The SFE is known to mainly depend on the alloy composition and test temperature.

Traditionally, the TWIP steels based on element systems of Fe-Mn-C [7] and Fe-Mn-Al-Si [8] are most common in the literature publication. To further improve the overall performance, the element addition of Ti [9] or Nb [10] is used to achieve the strengthening effect. Scott and Allain designed a new type of TWIP steel in the Fe-Mn-C system [11, 12], whose tensile strength can increase to 1000 MPa, and the elongation decreased to 50%. The secondary twins and serrated flow were clearly observed in the deformed matrix of Fe-22Mn-0.6 C
steel. As for the Fe-25Mn-3Si-3Al TWIP steel with higher stacking fault energy (SFE) value, the secondary ε-martensite/deformation twins is hindered from formation during deformation [13]. Delayed fracture [14], associated with hydrogen segregation [15], ε-martensite transformation, and twinning intersection [16], has been experimentally confirmed in Fe-Mn-C TWIP steel recently.

Although the higher contents of C, Al, and Si can guarantee the TWIP effect and mechanical properties, the relatively low yield strength, low weldability, and flowability have hampered the industrial application of TWIP steel. Thus, it is proposed to improve the mechanical properties of TWIP steel via the addition of vanadium (V) through the grain refinement and precipitation hardening [17]. Compared with the Ti or Nb elements, the V precipitation can be re-dissolved during the temperature increase. Thus, it can lead to a more pronounced grain refinement effect through the recrystallization annealing [18]. Besides, the V additions can also improve the tensile strength and yield strength, which can be achieved through the higher strain hardening rate at the deformation stage of the tensile test. Meanwhile, it was also reported that the precipitation of V₄C₃ carbides could effectively affect the nature of the stacking fault [17].

However, there are few reports about the TWIP steel with vanadium (V) and the effect of V on the microstructure and mechanical properties. In this work, the author tries to reduce the defects of traditional TWIP steel and obtain superior performance by adding alloying elements V in the Fe-Mn-C system. Studies about the influence of the annealing process on the microstructure and mechanical properties of TWIP steel with the addition of V were made. The microstructure and mechanical properties of TWIP steel after different annealing treatments were investigated by static tensile test, OM, TEM, and EBSD.

2. Experimental procedures

The chemical composition of the TWIP steel used in the investigation is presented in table 1. It was designed based on the model of Allain [19] to ensure a full austenite microstructure at room temperature. The TWIP steel was firstly melted in a vacuum induction furnace and then cast into a 300 mm × 150 mm × 150 mm slab. The billet was heated to 1150 °C, soaked for 30 min, and then underwent the hot-rolling process for 3 to 5 times till the thickness was reduced to 3 mm. The final rolling temperature is equal to or higher than 850 °C. The oxide film on the surface of hot-rolled plates was then removed by an acid solution of 16% HCl, followed by cold rolling with the thickness reduction of around 50% and 60%.

The post-annealing treatments were conducted on the prepared TWIP steels at the temperatures of 800 °C, 900 °C and 1000 °C for 30 min, followed by air cooling. The tensile test samples were prepared along the rolling direction. All the tensile tests were performed in a CMT5305 tensile test machine controlled by computer at a constant strain rate of 3 mm min⁻¹ at room temperature. Metallography specimens and EBSD samples were prepared along the longitudinal section. The surface stress layer was removed by electrolytic polishing. Nikon LV150 optical microscope (OM) and electron backscatter diffraction (EBSD) was used to analyze the microstructure, grain orientation, etc. DLMAX-2550 x-ray diffractometer with Cu Kα-beam was used to identify the phase structure, with a scanning speed of 1° min⁻¹. The microstructure was further investigated via a JSM-2010F SEM and JSM-6700F TEM to take advantage of a higher spatial resolution. The samples were mechanically polished to 60 μm in thickness, and the TEM foils were electrolytically thinned to electron transparency by using STRUERS thinning facility and a 5 vol% perchloric acid 95 vol% acetic acid solution at 40 V DC.

3. Results and discussion

3.1. Microstructure evolution

The XRD investigation was made on the TWIP steel after the annealing treatment at 800 °C and 1000 °C. Meanwhile, the fractured TWIP steel annealed at 1000 °C after the tensile test was also examined by XRD as a comparison. The XRD spectra of the abovementioned samples were given in figure 1. It can be seen that both the annealed TWIP steel samples are composed of a full austenite phase. As for the fracture sample after the tensile test, a similar phase composition can be observed. Afterward, the phase composition obtained by XRD tests is used to assist the EBSD analysis.
EBSD investigation was then made to further enhance the understanding of grain orientation and size distribution of the TWIP steel after different annealing treatments. The IPFs (Inver polar figures) via EBSD of the annealed samples at different temperatures were presented in figures 2(a)–(c). The optical micrograph of the steel after deformation annealed at 1000 °C is given in figure 2(d). It can be observed that the grain size increases with the increasing annealing temperature, and the equiaxed austenite structure was produced at three different annealing temperatures as a result of recrystallization. It is evident that the austenite grain contains a large number of annealing twins. In order to observe the volume fraction of annealing twins, the grain boundary distribution of TWIP steel was evaluated via EBSD analysis. Generally, the volume fraction of the annealing twins is obtained according to the Coincidence Site Lattices (CSLs) of $\Sigma 3$, whereas the calculation was achieved by running the software of EBSD OIM analysis provided by EDAX. The $\Sigma 3$ boundaries may also be twin boundaries exhibiting angle/axis boundary geometry $60^\circ/\langle 111 \rangle$ [20]. The grain boundaries of TWIP steel after annealing at 1000 °C is given in figure 3(a), where the $\Sigma 3$ boundaries are marked in red and $\Sigma 9$ boundaries are in

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**Figure 1.** XRD spectra of the TWIP steels annealed at 800 °C and 1000 °C, as well as the 1000 °C-annealed sample after the tensile test.

**Figure 2.** EBSD maps of the TWIP steels annealed at different temperatures: (a) 800 °C, (b) 900 °C, (c) 1000 °C, and (d) optical micrograph of the steel after deformation annealed at 1000 °C.
black. As shown in figure 3(b), the volume fraction of the annealing twins (Σ3) increases from 31% to 39% as the annealing temperature increases from 800 °C to 1000 °C. As shown in figure 3(b), the average grain size increases from 3.66 μm, 5.04 μm to 26.8 μm as the annealing temperature increases from 800 °C, 900 °C to 1000 °C. Figure 2(d) shows a detailed view of deformation twins, which exhibited symmetrical arrangement around the twin axis. Besides, it can also be noticed that the volume fraction of Σ9 is decreasing at the higher annealing temperature. The grain size and misorientation angle both increase with the increase of annealing temperature. Thus, the High angle grain boundary (HAGB) with higher grain interfacial energy tends to move toward the grain boundaries with lower interfacial energy, which results in lower volume fraction of Σ9 boundaries [20].

The TEM microstructures of the TWIP steels under different annealing treatments are shown in figure 4. Figures 4(a) and (b) show that a large number of twin bundles in the TWIP steels annealed at 800 °C and 1000 °C, which grew in the same direction as the austenite grain. As shown in figure 4(a), the multiple-twinned beam is composed of a twinned region, and the upper-right corner of the selected area is the electron diffraction result. It can be suggested that several twin-bundles were generated during the annealing process, some of which were connected to form larger twin regions. It can be seen that the twin bundle spacing of the TWIP steel annealed at 1000 °C is smaller than that of 800 °C. As shown in figures 4(c) and (d), secondary phase particles with V or Ti, which enriched the austenite grains within and at boundaries, were observed annealing at temperatures of 800 °C and 1000 °C. Based on image analysis by ImageJ, it is able to quantitatively obtain the size variation of the secondary phase particles in the TEM image. As shown in figures 4(c) and (d), the precipitate particle experienced an evident growth at higher annealing temperature. Based on the image analysis, the average size of the second phase increases to 0.3 μm at 1000 °C from 0.05 μm at 800 °C. Figure 5 shows the deformation twins within the TWIP steels annealed at 800 °C and 1000 °C after tensile deformation. A lot of long bundles of parallel and staggered arranged deformation twins can be observed. As shown in figure 5(a), a large number of deformation twins with various widths and similar orientation appear at the annealing temperatures of 800 °C. Figure 5(b) shows four different sets of orientation twins with an angle of about 60° (T1, T2, T3, and T4). It can also be observed that most of the deformation twins formed within the austenite, and ended at the grain boundaries (T1, T3, and T4). Figure 5(c) shows multiple twinned regions within the TWIP steel annealing at 1000 °C after deformation, and the broader twin-bundles were composed of a few nanometer-scale micro twins. Consequently, the final morphology of the deformation twins was formed by several twin-bundles, which grew and developed the final coherent with twin regional components. Twins observed in the metallographic microscope are composed of several twin-bundles of the twin’s region (figure 2(d)). Figure 5(d) shows the morphology of deformation twins with a different orientation - namely, T1 and T2. T2 was a continuous twin, but T1 was truncated by T2 with different thicknesses. So, it can be inferred that they were gradually formed. Allain suggested that the deformation twins formed by Shockley imperfect dislocation slip in the [111] plane and a/6 (112) direction. The deformation twins started to form when the stress reached the critical nucleation stress, which was composed of extended stacking faults and dislocation loops [19]. The micro-stress distribution will affect the nucleation and growth of deformation twins, thus resulting in different thicknesses of the twinned region in a twinning system. Compared with figure 5, much more deformation twins can be found in the TWIP steel annealed at 1000 °C, and the space between twin-bundles was less than that corresponding one at 800 °C. So, the TWIP steel has better mechanical properties after annealing treatment at 1000 °C.
Generally, the deformation twins are formed by the twin dislocation movement overcoming the critical stress in the first place. Any factors that impede dislocation movement will cause an increase in critical stress to form deformation twins [19]. Therefore, the critical stress to produce deformation twins strongly depends on the magnitude of the grain size, which meets the relationship of Hall-Petch [19, 21]:

\[ \sigma_T = \sigma_{T0} + K_T d^{-A} \]

where \( \sigma_T \) is the critical stress to produce deformation twins, \( \sigma_{T0} \) is the lattice friction, \( K_T \) and \( A \) (1/2 \( \leq A \leq 1 \)) is the constant, and \( d \) is the grain diameter. It can be seen the critical stress to produce deformation twins decreases with increasing grain size, and the twinning will stop as the grain size is sufficient low. \( \sigma_T \) is found to decrease as the austenite grain size increases from 3.66 \( \mu m \) to 26.8 \( \mu m \). Thus, it formed much more deformation twins to gain the best mechanical properties annealing at 1000 \( ^\circ \)C.

3.2. Mechanical properties

The uniaxial tensile test was carried out on the TWIP steels annealed under different temperatures, and the engineering stress-strain curves are shown in figure 6. As expected, the strength decreases with the increasing...
annealing temperature. However, the tensile elongation to fracture increases as the annealing temperature increases up to 1000 °C. As for the sample annealed at 1000 °C, the ultimate tensile strength reaches 1136 MPa, and the elongation is approaching 70%. The product of strength and elongation is exceeded by $7 \times 10^4$ MPa·%.

Serration features can be observed on the stress-strain curves of the TWIP steels under different annealing processes. It is also interesting to notice that the amplitude of serration is increasing at a higher strain value, which is notably different from the end region of the curves of 900 °C and 1000 °C samples. The occurrence of serrations on true stress-strain curves of high manganese austenitic steels is well known as the dynamic strain aging mechanism or 'Portevin-Le Chatelier' effect [22]. The serration-shaped plastic flow is a result of localized

**Figure 5.** TEM images of the deformation twins after tensile deformation: (a), (b):800 °C; (c), (d):1000 °C.

**Figure 6.** Engineering stress-strain curves of TWIP steels processed at different annealing temperatures.
plastic deformation and the nucleation of the localized deformation band. It can also indicate the dislocation multiplication during the tensile test to maintain the constant strain rate [23]. The prominent slopes in the elastic deformation stage show that the TWIP steels have high elastic modulus, and the plastic deformation occupied large deformation space where no obvious yield point exists.

Curves of strain hardening rate $\frac{d\sigma}{d\varepsilon}$ and work hardening exponent $n = \frac{d\sigma}{d\varepsilon} \cdot \frac{1}{\varepsilon}$ are shown in figures 7(a) and (b), respectively. These curves were both obtained from the derivation of true $\sigma - \varepsilon$ curve after smooth treatment. As shown in figure 7(a), remarkably high initial strain hardening rates were obtained at all of the three annealed samples. The curve of strain hardening with true strain can be divided into three stages for the annealing temperature of 1000 °C. The value of $d\sigma/d\varepsilon$ is higher than 800 MPa, while a true strain is below 0.5. During the deformation, the strain hardening rate evolves according to different stages. After a rapid drop of strain hardening rate to the value of 3000 MPa in stage I where the true strain ranges from 0 to 0.06, a platform appears for the stage II where the true strain ranges from 0.06 to 0.25. It results in the deformation twins have formed [19, 21]. The number of deformation twins increased with the increasing of true strain at this stage. Finally, for true strain higher than 0.25, the strain hardening rate decreases again (stage III) till the specimen ruptured, which shows that the formation rate of deformation twins decreased [19]. For TWIP steels annealed at temperatures of 800 and 900 °C, it has the same tendency as 1000 °C-sample, namely the strain hardening rate rapidly decreases with the true strain increasing at the stage I. While the stage II becomes shorter, the platform disappeared at this stage. From figure 7(a), also it found that the strain hardening rate decreases more slowly with the increasing annealing temperature in stages II and III. Meanwhile, the austenite grain size increased with the increasing temperature (figure 2). It can be inferred that the large grain size can guarantee a continuing hardening behavior in the deformation process and a higher hardening ability.

Figure 7(b) shows the relationship between work hardening exponent $n = \frac{d\sigma}{d\varepsilon} \cdot \frac{1}{\varepsilon}$ and true strain $\varepsilon$ under different annealing processes. The relationship between true stress and true strain does not follow the Hollomond relation [19]. The value of $n$ increases and maintains at a high level with the increase of high true strain. An identical tendency appears for these three curves when $\varepsilon$ is less than 0.7. It increases with the increasing annealing temperature, ranging from 800 °C to 1000 °C, in order to obtain the maximum values of 0.53, 0.62 and 0.70 for $n$, respectively. From the analysis above, a conclusion is drawn that at higher annealing temperature, the greater capacity of even distribution of strain exists due to the hardening, and the ability to resist necking during deformation is also better. As for the sudden increase of the value $n$ during the late period of deformation of the curve (1), it may be due to the serration characteristics of the engineering strain-stress curve (see figure 6). The serration behavior observed in the strain-stress curve can also indicate the dislocation multiplication during the tensile test to maintain the constant strain rate. The increasing dislocation density will further lead to the increase of work hardening rate [24, 25], which can be seen as the sudden increase of work hardening exponent $n$. Besides, a sudden fracture happened when the stress increased, resulting in a deviation from the general law at the curve fitting.

3.3. Fracture morphology analysis

Figure 8 shows the fracture morphology at the different annealing temperatures. Equiaxed dimples with large depth and diameter existed under the three different annealing processes. The secondary particles existed at the bottom of the dimples, and the inserted image in figure 8(b) shows the magnified morphology of the boxed dimple areas. It can be speculated as the vanadium or titanium compounds from figure 4. Due to the different
mechanical properties between the secondary particles and the matrix, microporous cores form in the process of plastic deformation along the boundary of the secondary phase particles or within the secondary phases. Under the effect of stress, the core of these micro-cracks gradually grows up, and microscopic holes in the connection section become thinner until the final fracture with the increasing plastic deformation. There were lots of micron grade secondary dimples that formed for the reason of the C-Mn atom pair and the dynamic strain aging between and within the big simples (figure 8) [26]. The nanoscale dimples originated in and between the big dimples make micron-sized dimples together [19]. This is also one of the reasons the TWIP steel did not appear the necking in the process of the tensile test.

4. Conclusions

1. It is evident that the austenite grain size increases with the increasing annealing temperature from 800 °C, 900 °C to 1000 °C. However, the tensile strength decreases and the elongation decreases at higher annealing temperature, whereas the product of strength and elongation is exceeding $7 \times 10^4$ MPa-% at 1000 °C.

2. The investigated TWIP steel has a higher initial work-hardening rate. The curves of strain hardening rate turn to three stages from two stages with increasing annealing temperature. The strain hardening index ($n$) has the same change tendency under different annealing processes when $\varepsilon < 0.07$. With the true stain increasing, the $n$ value reached the maximum value and can last at a large strain, which is 0.53, 0.62, and 0.70.

3. The volume fraction of annealing twins increased with the increasing temperature, which was up to 39% at 1000 °C. The secondary phase particles of V or Ti elements existed under different annealing processes, which increased in size with the increasing temperature.

4. Only some of the austenite grain exhibited twin systems with different orientations in some after tensile deformation. The twin spacing decreased with increasing temperature, thus producing a sustained work hardening effect, which enhanced the "TWIP" effect.
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ORCID iDs

Chaoyue Chen @ https://orcid.org/0000-0003-3696-7769

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