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Deformation behaviour and finite element method modelling of TWinning induced plasticity (TWIP) steel

Ching-Tun Peng
University of Wollongong

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Deformation Behaviour and Finite Element Method Modelling of TWinning Induced Plasticity (TWIP) Steel

A thesis submitted to the University of Wollongong as fulfilment for the degree of

Doctor of Philosophy

By
Ching-Tun Peng

School of Mechanical, Materials and Mechatronic Engineering
University of Wollongong, Australia
2014
Declaration

I, Ching-Tun Peng, declare that this thesis, submitted in fulfilment of the requirements for the award of Doctor of Philosophy, in the school of Mechanical, Materials and Mechatronics Engineering, University of Wollongong, Australia, is wholly my own work unless otherwise referenced or acknowledged. The document has not been submitted for qualifications at any other university of academic institution.

Ching-Tun Peng

August, 2014
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I would like to give my sincere thanks to my supervisors, A/Prof. Huijun Li and Dr. Mark. D. Callaghan for their constant patience, support and guidance. Thanks are also due to R. de Jong and G. Tillman at the University of Wollongong for assistance with Gleeble 3500 experiments and metallography and the use of facilities within the UOW Electron Microscopy Centre is acknowledged as well.

I am also grateful to Mark D. Callaghan, Kun Yan, Klaus-Dieter Liss, and D. Carr for participation in high-energy X-ray diffraction experiments at the European Synchrotron Radiation Facility. Thanks are also given to Prof. Tuan D. Ngo and Dr. Priyan A. Mendis for the Split Hopkinson Pressure Bar experiments conducted at the University of Melbourne and to Dr. Chang-Ho Choi for providing the material and the Explosion Bulge Testing conducted at Defence Science and Technology.

The supports from Mr. Mao Liu and Dr. Cheng Lu for the Crystal Plasticity Finite Element Method modelling and the help of nano-indentation experiments done by Mr. Yong Sun are also deeply appreciated.

Finally, I would like to thank my family for their patience, encouragement and endless love.
Publications during the PhD course

1. **Ching-Tun Peng**, Mark D. Callaghan, Huijun Li, Kun Yan, Klaus-Dieter Liss, Tuan D. Ngo, Priyan A. Mendis and Chang-Ho Choi (2013). “On the Compression Behaviour of an Austenitic Fe-18Mn-0.6C-1.5Al Twinning-Induced Plasticity Steel”, *steel research international* 84: 1281-1287.


3. **Ching-Tun Peng**, Mark D. Callaghan, Huijun Li, Kun Yan, Klaus-Dieter Liss, Tuan D. Ngo, Priyan A. Mendis and Chang-Ho Choi. “On the Compression Behaviour of an Austenitic Fe-18Mn-0.6C-1.5Al Twinning-Induced Plasticity Steel”. In: *15th International conference on Advances in Materials and Processing Technologies*, September 23-26, 2012, Wollongong, Australia, 11736

5. Chang-Ho Choi, Ching-Tun Peng, Brian F. Dixon, Huijun Li. “Pre-blast strengthening of Fe-18Mn-0.6C-1.5Al TWIP steel”, manuscript accepted by steel research international.


7. Ching-Tun Peng, Mark D. Callaghan, Huijun Li. “Post-deformation Microstructure and Texture Characterisation of Fe-18Mn-0.6C-1.5Al TWIP steel”, manuscript submitted.

Abstract

TWinning Induced Plasticity (TWIP) steels have been placed as promising materials for the next generation of auto-related materials as well as the military applications due to their exceptional energy absorption ability. The present work investigates the compression and blast behaviour of Fe-18Mn-1.5Al-0.6C TWIP steel using various diffraction techniques and single crystal plasticity finite element method modelling.

The compression behaviour of TWIP steel has been investigated at various strain rates with different strains, including a Split Hopkinson Pressure Bar (SHPB) apparatus, and the results exhibited outstanding strain hardening. This unique feature is ascribed to mechanical twinning occurring during deformation which was proven by microstructural and crystallographic characterization and analysis. Mechanical twinning heavily generate during straining while slip makes a contribution. Tensile deformation favours twinning activity as compared to compression. An Explosive Bulge Test (EBT) was successfully conducted on the TIWP steel which provides a feasible way of strengthening the material and the grain refinement, caused by this pre-blast technique, was the main strengthening mechanism.
This is the first time a Crystal Plasticity Finite Element Method (CPFEM) model, based on single crystal assumption, was constructed in Abaqus environment with user material subroutine to simulate the course of nano-indentation of a TWIP steel. The crystal orientation, being necessary input parameters, of the TWIP steel was obtained from Electron BackScattered Diffraction (EBSD). Three unknown self hardening parameters were determined by fitting the experimental and simulated load-displacement curves.
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<th>Description</th>
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<tbody>
<tr>
<td>AHSS</td>
<td>Advanced High Strength Steel</td>
</tr>
<tr>
<td>TWIP</td>
<td>TWinning Induced Plasticity</td>
</tr>
<tr>
<td>SHPB</td>
<td>Split Hopkinson Pressure Bar</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron BackScattered Diffraction</td>
</tr>
<tr>
<td>CPFEM</td>
<td>Crystal Plasticity Finite Element Method</td>
</tr>
<tr>
<td>UMAT</td>
<td>User Material Subroutine</td>
</tr>
<tr>
<td>UHSS</td>
<td>UltraHigh Strength Steels</td>
</tr>
<tr>
<td>HSS</td>
<td>High Strength Steel</td>
</tr>
<tr>
<td>IF</td>
<td>Interstitial-Free</td>
</tr>
<tr>
<td>AHSS</td>
<td>Advanced High Strength Steels</td>
</tr>
<tr>
<td>DP</td>
<td>Dual Phase</td>
</tr>
<tr>
<td>CP</td>
<td>Complex Phase</td>
</tr>
<tr>
<td>PM</td>
<td>Partly Martensite</td>
</tr>
<tr>
<td>TRIP</td>
<td>TRansformation Induced Plasticity</td>
</tr>
<tr>
<td>HMS</td>
<td>High Manganese Steels</td>
</tr>
<tr>
<td>SFE</td>
<td>Stacking Fault Energy</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission Electron Microscopy</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic Force Microscopy</td>
</tr>
<tr>
<td>XRD</td>
<td>X-Ray Diffraction</td>
</tr>
<tr>
<td>ECCI</td>
<td>Electron Channelling Contrast Imaging</td>
</tr>
<tr>
<td>SIP</td>
<td>Shear band Induced Plasticity</td>
</tr>
<tr>
<td>DSA</td>
<td>Dynamic Strain Aging</td>
</tr>
<tr>
<td>SSA</td>
<td>Static Strain Aging</td>
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<tr>
<td>Abbreviation</td>
<td>Definition</td>
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<tr>
<td>PLC</td>
<td>Portevin-Le Chatelier</td>
</tr>
<tr>
<td>DIC</td>
<td>Digital Image Correlation</td>
</tr>
<tr>
<td>HDF</td>
<td>Hydrogen Delayed Fracture</td>
</tr>
<tr>
<td>ANN</td>
<td>Artificial Neural Network</td>
</tr>
<tr>
<td>EBT</td>
<td>Explosive Bulge Test</td>
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<tr>
<td>ESRF</td>
<td>European Synchrotron Radiation Facility</td>
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<td>OM</td>
<td>Optical Microscope</td>
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<td>TD</td>
<td>Transverse Direction</td>
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<td>HAGBs</td>
<td>High Angle Grain Boundaries</td>
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<td>ODF</td>
<td>Orientation Distribution Function</td>
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<tr>
<td>UFG</td>
<td>Ultra-Fine Grain</td>
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<td>SPD</td>
<td>Severe Plastic Deformation</td>
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<td>VPSC</td>
<td>ViscoPlastic Self-Consistent</td>
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<td>martensite start temperature</td>
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2. Symbols

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<thead>
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<tr>
<td>Espec</td>
<td>specific energy absorption</td>
</tr>
<tr>
<td>$\gamma$</td>
<td>(fcc) austenite</td>
</tr>
<tr>
<td>$\epsilon$</td>
<td>(hcp) martensite</td>
</tr>
<tr>
<td>$\alpha'$</td>
<td>(bcc) martensite</td>
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<tr>
<td>$\Gamma_{SFE}$</td>
<td>Stacking Fault Energy</td>
</tr>
<tr>
<td>$R_{p0.2}$</td>
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<tr>
<td>$R_m$</td>
<td>tensile strength</td>
</tr>
<tr>
<td>$\varepsilon_{un}$</td>
<td>uniform elongation</td>
</tr>
<tr>
<td>$\varepsilon_f$</td>
<td>total elongation</td>
</tr>
<tr>
<td>Symbol</td>
<td>Description</td>
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<tr>
<td>--------</td>
<td>-------------</td>
</tr>
<tr>
<td>$\Gamma$</td>
<td>austenitic SFE</td>
</tr>
<tr>
<td>$\rho$</td>
<td>atomic density</td>
</tr>
<tr>
<td>$\Delta G_{\gamma \rightarrow \varepsilon}$</td>
<td>changes in the Gibbs free energy</td>
</tr>
<tr>
<td>$\sigma_{\gamma / \varepsilon}$</td>
<td>surface energy</td>
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<td>$\Sigma 3$</td>
<td>first order twin boundaries</td>
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<td>second order twin boundaries</td>
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<td>$f_e(g)$</td>
<td>texture intensities</td>
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<td>$C_{11}, C_{12}, C_{44}$</td>
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<td>initial hardening modulus</td>
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<tr>
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Chapter 1

Introduction

With increasing oil price and stricter gas emission standard, auto-buyers are eager for vehicles with more environmental-friendly, economic efficiency, better performance, and safer bodywork, which encourages auto manufacturers to develop novel automobiles. Without materials, there is no engineering. Therefore, as being the main supplier of the auto body materials, steelmakers are driven to investigate advanced high strength steels with light-weight and high strength in order to satisfy those demands while they are also facing increasing challenges from other materials, such as aluminium, magnesium and plastics.

As being a new class of Advanced High Strength Steel (AHSS), high-manganese austenitic TWinning Induced Plasticity (TWIP) steels, are of great interests in the field of automotive industry where their exceptional combination of mechanical properties (strength and ductility) provide better crashworthiness. These extreme tough and particularly ductile materials are suitable for the road or the railway, making vehicles stronger and safer. In
the case of an accident, this “super-tough” material absorbs a large amount of energy under extreme impact loading, as a result, the safety of passengers is greatly enhanced.

In this work, a new member of High Manganese Steels (HMS), Fe-18Mn-1.5Al-0.6C TWIP steel was chosen to conduct current study since this specific materials only been brought to the research field 5 years ago and the literature is lacking of certain deformation data as well as the self hardening parameters. Therefore, this work investigated its compression behaviour at various strain rates with different level of strain by adopting both Split Hopkinson Pressure Bar and Gleeble system. The mechanical properties, including stress-strain curves, yield stress, working-hardening rate and hardness were successfully obtained. The microstructure was analysed by optical microscopy and Scanning Electron Microscopy (SEM), furthermore, the crystallographic features of the TIWP steel was characterized by Electron BackScattered Diffraction (EBSD) technique as well as the ID15B beamlines located at the European Synchrotron Radiation Facility.

A Crystal Plasticity Finite Element Method (CPFEM) model was built in Abaqus environment with the User Material Subroutine (UMAT) to simulate
the nano-indentation process. The vital input parameters, actual crystal orientation, of UMAT were obtained from EBSD technique. Other unknown input parameters could be acquired by fitting method, which is matching the experimental and simulated load-displacement curves. The three self-hardening parameters were successfully determined. The simulated surface profile, pole figure and lattice rotation angles were developed by post processing through Matlab.
Chapter 2

Literature review

Steel, iron-carbon alloys which consist mostly of iron, is the most widely used material. The history of steel utilization can track back to thousands of years ago. Among all kinds of metals, iron are the most exploited in quantities owing to the abundant quantities of iron mineral within earth’s crust. The widespread use of steels is due to their variety of properties, but their success in engineering is resulted from the good combination of strength, ductility and ease of manufacture. Steels are the most versatile engineered materials. They are especially important as engineering and load-bearing construction materials. Most buildings, bridges, automotive, tools, and numerous other applications make use of ferrous alloys. Steels can be classified by the strength values. The terminology UltraHigh Strength Steels (UHSS) is applied as the yield strength is greater than 550 MPa. While the yield strength ranges from 210 to 550 MPa, those steels are named High Strength Steel (HSS). Low strength steels have yield strengths lower than 210 MPa.
The strength of steel is derived from the microstructure and resultant property relationship, which is, in turn, influenced by both the composition, as well treatment during processing. The conventional mechanisms to increase the strength in steel such as solid solution hardening or precipitation hardening are accompanied by a noticeably inferior formability. Conventional high strength steels were produced by adding, for instance, Nb, Ti, V, and/or P in Interstitial-Free (IF) or low carbon steels. These steels have been widespread utilized for weight reduction due to the relatively simple manufacturing conditions.

However, with increasing demands in the field of automotive industries, the legal requirement (stricter CO₂ target emission and crashworthiness) and customer expectation (design, low cost, performance, better fuel efficiency, corrosion, etc.) are driving forces for the development of new materials. Fuel efficiency and safety issues are always the primary challenges in the design of automobile. The demands for making cars both more environmental friendly and greater crashworthy have forced steelmakers, who are major materials suppliers of auto manufacturers, to develop novel materials with improved strength and formability. In the past few decades, an increasing number of researchers have devoted to both, for short term solution, improvement of the existing materials and, for the long term strategy, design
of novel materials. The introduction of a new family of steels with a microstructure consisting of at least two different components has led to a superior level of strength without sacrificing ductility. As shown in Figure 2-1 Advanced High Strength Steels (AHSS) and high manganese steels are categorized within this group. These multiphase steels offer very attractive combinations of strength and ductility resulting from the coexistence of the different microstructural components and their mutual interactions and the outstanding mechanical properties can be adjusted by alloying process [1, 2].

Figure 2-1 Materials-Strength vs Formability [2].
AHSS are determined based on their microstructural characteristics. These multiphase steels contain martensite, bainite and retained austenite and offer extraordinary strength-ductility relationships. Therefore, they are of primary interests for vehicle applications. AHSS includes Dual Phase (DP), Complex Phase (CP), Partly Martensite (PM) and TRansformation Induced Plasticity (TRIP) steels. DP and TRIP steels have been widely used in various automotive parts in recent times. Due to the relative ease of manufacture, the application of DP steels is fairly popular among automakers. TRIP steels, providing the highest combination of strength and elongation among AHSS, is for high level energy absorption purpose.

2.1 High manganese steel

High Manganese Steels (HMS), a family of austenitic steels with high manganese contents (usually 15-30 wt.% Mn) recently has been gaining great attention for automotive use owing to their exceptional combination of tensile strength and ductility. The first found high manganese steels can be traced back to the 19th Century, by the work of Hadfield. These steels were found to display good wear resistance, toughness and excellent ductility despite exhibiting high hardness. Moreover, the manufacturing cost is reduced with alloying concept while comparing with other conventional or
new high strength steels. As different mechanism occur during deformation, these steels are divided into TRansformation Induced Plasticity (TRIP) steels and TWinning Induced Plasticity (TWIP) steels. In which the TWIP steels were found that deformation twins inhibit gliding dislocations resulting in increasing work-hardening rate [3]. The controlling of the manufacturing condition of those high manganese steels is the key to produce the optimal strength-ductility.

The mechanism of the high Mn steel is the interaction between dislocation gliding and other possible deformation Even though the dislocation glide is the dominant mechanism of the plastic deformation, the TRIP effect or the TWIP effect can occur depending on the chemical composition and the condition of the treatment. The stacking fault energy is the key factor to the phase transformation during deformation mechanisms, such as phase transformation and mechanical twinning. In the course of deformation, those phenomena are competing with one another, providing a means of developing materials with extraordinary combination of strength and ductility. Due to the excellent energy observing feature, these newly developed alloys are potential candidates for civil and military usage.
Since a decade ago, steel manufacturers have been intensively investigating the development of ultra-high strength high-Mn based austenitic steels with TWIP effect due to their outstanding strength-ductility combination. These steels show far better mechanical properties and formability than other ferrous products. Frommeyer’s group began to study high Mn steels and disclosed their promising combination of mechanical properties (range from 650-900MPa ultimate tensile strength and 60-95% elongation) which render the higher value of specific energy absorption, Espec, of 0.5 J/mm³, compared with those of conventional deep-drawing steels, such as interstitial-free steels, bake hardening steels and thermo-mechanically processed steels, whereby $0.16 \leq \text{Espec} \leq 0.25 \text{ J/mm}^3$ [4]; therefore, these materials have drawn significant attention in the field of materials engineering. Deformation temperature, strain rate and chemical composition are factors influencing the tensile strength and elongation. Their super tough and super ductile characteristics, therefore, have provided a great potential in the automotive application of many structural components as shown in Figure 2-2, such as chassis, engine compartment, bumpers and side doors[3]. The automotive crashworthiness can be significantly enhanced by implementing these novel materials. Weight reduction can also be achieved for the purposes of better fuel efficiency and meeting the stricter gas emission standards. A remarkable
amount of researchers have been devoted to the development of high Mn TWIP steels [3-11]. Among them, Fe-(~25)Mn-(~3)Si-(~3)Al [4, 6, 12] and Fe-(~22)Mn-(~0.6)C[13, 14] were intensively investigated.

Figure 2-2 Automotive applications of high-Mn TWIP steel (courtesy by ArcelorMittal Flat Carbon Europe).

2.1.1 Deformation modes in high manganese steels

Other than the TWIP effect, another deformation mechanism, TRIP effect could also be observed from these high manganese steels [12, 15-17]. Results indicated that the TWIP effect may make more contribution to the plasticity
of the materials, while the TRIP effect is beneficial to the strength of the materials [14, 15]. To briefly address the prime difference between high manganese TRIP and TWIP steels, the later, TWIP steels have no phase transformation during cooling or deformation, but the orientation of part of the austenite will change due to mechanical twinning resulting in excellent strength and ductility. As for as TRIP steels are concerned, the austenite is stable during cooling but not under mechanical stress, i.e. phase transformation happens when the alloys are loaded [3]. When an austenitic high-Mn TRIP steels plastically deform as strain-induced, martensite could be formed as $\gamma - (fcc)$ austenite $\rightarrow \varepsilon - (hcp)$ martensite or in two-steps reaction $\gamma - (fcc)$ austenite $\rightarrow \varepsilon - (hcp)$ martensite $\rightarrow \alpha'$ - (bcc) martensite [18]. Depending on various chemical compositions and the processing procedures, these two deformation mechanisms can either appear individually or coexist [13, 19]. Other than phase transformation, both dislocation slips and mechanical twinning are two essential mechanisms accounting for plastic deformation.

A stack of close-packed layers of atoms arranged in a periodic sequence is known as a crystal. However, errors may exist in the sequence, called stacking faults. Like all other defects, a stacking fault results in change in energy, named Stacking Fault Energy (SFE), denoted $\Gamma_{SFE}$ (J/m$^2$). It is well
known that SFE is responsible for the different deformation behaviours of the austenite as shown in Figure 2-3. SFE dominates the ease of cross-slip of dislocations and thus different mechanisms can be stimulated at various stage of deformation. Therefore, mechanical twinning tendency of the austenitic structure can be roughly predicted by SFE. In other words, its value determines the main deformation mechanism in the steels. With a decreasing SFE value, wider stacking faults are formed whereas cross-slip become more difficult to form, and the deformation mechanism changes from dislocations slips to partial slips, to mechanical twinning and eventually to phase transformation [20].
Figure 2-3 Schematic presentation of the influence of SFE (temperature and composition) on the features in deformation of austenite [21].

Both deformation temperature and chemical composition are in charge of controlling SFE, therefore by adjusting the mechanical composition and deformation temperature, the stacking fault energy can be controlled to specific range where the kinetics of twinning are highest, twinning is the dominating deformation mechanism, and the high manganese steel reaches the maximum ductility and further result in extraordinary mechanical properties, which is proven empirically in Figure 2-4 and 2-5.
Figure 2-4 Dependence of Yield stress $R_{p0.2}$, tensile strength $R_m$, uniform elongation $\varepsilon_{un}$ and total elongation $\varepsilon_f$ on test temperature of Fe-20Mn-3Si-3Al TRIP steel [4].

Figure 2-5 True stress vs. true strain curves of the TRIP and TWIP steels; test temperature: 20°C, strain rate: $10^{-4}$s$^{-1}$ [10].
According to Remy and Pineau [22], mechanical twinning appears when the SFE is higher than 9 mJ/m$^2$ while the phase transformation occurs with SFE lower than 12 mJ/m$^2$. The results of Oh et al. [23] show that the minimum SFE needed for deformation twins is 18 mJ/m$^2$ while $\varepsilon$-martensite formation requires lower value [21]. Allain et al. [24] proposed a calculation which indicates that mechanical twins form while SFE between 12 and 35 mJ/m$^2$ whereas martensitic transformation can be formed when the SFE does not exceed 18 mJ/m$^2$. Because of the diverse chemical compositions, these literature data have come to a reasonably good agreement. Olson and Cohen (1976) proposed an equation to calculate the SFE

$$\Gamma = 2\rho \Delta G^{\gamma \rightarrow \varepsilon} + 2\sigma^{\gamma / \varepsilon} \tag{2.1}$$

Where $\Gamma$ is the austenitic SFE, $\rho$ is the atomic density of a close packed plane in face cubic centre structure, $\Delta G^{\gamma \rightarrow \varepsilon}$ is the changes in the Gibbs free energy of the austenite to $\varepsilon$-martensite phase transformation, and $\sigma^{\gamma / \varepsilon}$ is the surface energy between austenite and $\varepsilon$-martensite phases. Therefore, the SFE of the Fe-18Mn-0.6C-1.5Al TWIP steel used in current work is calculated as 33 (mJ/m$^2$) [25].
High manganese steels are decomposed into several categories namely Fe-Mn-Si-Al, Fe-Mn-C, Fe-Mn-C-Al systems.

2.1.2 Fe-Mn-Si-Al system

Deformation mechanism and mechanical properties of high Mn steels, containing 15~30 wt. % manganese, with addition of Al (2~4 wt. %) and Si (2~4 wt. %) has been systematically investigated with tensile tests by Grässel and Frommeyer in detail [4]. When the manganese concentration is 15 wt. %, only TRIP is observed. Both TRIP and TWIP effects are found while manganese content is 20 wt. %. The major deformation mechanism is mechanical twinning with manganese content >25 wt. %. For a constant Mn content, alloys with both 3% Al and Si addition shows maximum tensile elongation. For example, a ~60% total elongation and a ~900MPa ultimate tensile are found on the Fe-15Mn-3Si-3Al steel. The highest elongation value is found to be about 95% from the Fe-25Mn-3Si-3Al, having the tensile strength of 650 MPa. The extraordinary combination of strength and elongation is believed to be resulted from the twinning effect. These steels with reduced specific weight (7.3 g/cm³), high specific energy absorption (0.5 J/mm³) and the exceptional impact toughness properties lead to a lighter but tougher car body [10]. The microstructure of TWIP steel can be observed
by optical and Transmission Electron Microscopy (TEM). Mechanical twinning enhances the work-hardening rate. In Figure 2-6 and 2-7, the thin fine twin lamellae inside the austenite matrix can be treated as obstacles impeding the mobility of dislocation, i.e. the twinning is responsible for maintaining the work-hardening rate by hindering the glide of dislocations. The amount of twinning and its morphology influence the mechanical properties.
Figure 2-6 Microstructure of TWIP steel: (1) Optical micrographs of typical TWIP steel (a) unstrained, (b) 18% strain, (c) 26% strain, (d) 34% strain [26].
Figure 2-7 Bright field transmission electron micrograph illustrating deformation twins [10].

Similarly, from a study of Zhen-li Mi et al., Fe-(16, 19, 23, 27)Mn-3Al-3Si steels have been investigated with tensile tests. The only different experimental variable is strain rate which is $10^2$ s$^{-1}$. With 23Mn and 27Mn, mechanical twins lead to the major contribution during deformation, on the other hand in both 16Mn and 19Mn, martensite transformation is dominant [27], which is corresponding with [4].
From Ding Hua’s research, both TRIP and TWIP phenomena are found in the Fe-23.8Mn-2.7Si-3.0Al steel, and the Fe-33Mn-2.93Si-3Al steel, only TWIP effect appears. Results indicate that TWIP effect may make more contribution to the increase of plasticity of the materials, while TRIP effect is beneficial to the strength of the materials [11]. With increasing manganese content, the strength decreased, while the plasticity of the material increased. This indicates that TRIP or TWIP effect plays the major role to the strength of these steels rather than solid solution strengthening.

Zhang et al. [28] combined Atomic Force Microscopy (AFM), EBSD and TEM to analyse the deformation mechanisms in both Fe-30Mn-4Si-2Al and Fe-30Mn-3Si-3Al with tensile tests. For Fe-30Mn-4Si-2Al, phase transformation happens in the onset of plastic deformation. As strain increases, some of the ε-martensite transform to twins. On the other hand, at the early stage of plastic deformation, planar and wavy dislocations were observed in the microstructure of Fe-30Mn-3Si-3Al. Mechanical twins generate and increase as strain increases. Rong-gang et al. found that only the martensitic transformation exists in a tensile tested Fe-14Mn-3Si-2Al. on the other hand, deformation twins were observed in Fe-25Mn-3Si-2Al. TWIP steels are sensitive to strain rate. As strain rate rise, the tensile strength increases significantly [17]. Ding Hao et al. [19] investigated deformation
modes and the tensile strain hardening behaviour with a Fe-18.8Mn-2.9Al-2.9Si. The strain hardening exponent remains constant in the beginning of plastic deformation and the TRIP effect was observed. While the strain rate ranges from 0.14 to 0.35, the strain hardening increases with increasing true strain. Mechanical twinning is the dominating deformation mechanism in this stage. Some TRIP effects are found with strain rate > 0.35 and both TWIP and TRIP coexists [19]. H. Idrissi et al. [29] studied that texture evolution of a Fe-19.7Mn-3.1Al-2.9Si. The specimens are tensile tested with various temperatures, room, 86°C and 160 °C. It was found that, at the beginning of plastic deformation, stacking faults appears moving from extrinsic to intrinsic. The deformation mode, simultaneously, alters from phase transformation to mechanical twinning.

From an investigation of Vercammen et al. [16], a cold rolled Fe-30Mn-3Si-3Al TWIP steel was studied by TEM and X-Ray Diffraction (XRD). Results conclude that different deformation mechanisms were observed with various cold rolling strains. At a low strain, 0.1, twinning was found in at least half of the grains. The volume fraction of twins increases with increasing strain. At the 0.21 strain, almost every single grain contains twins which leads to nano-scale lamella structure due to the small thickness,
50-100 nm, of the twins. Slip and mechanical twinning became active at higher strain level [16].

In a study of Ueji et al. [30], a Fe-31Mn-3Al-3Si TWIP steel with various grain size (1.8, 7.2, 49.6 μm) are investigated. The fine-grained steel (d = 1.8 μm) exhibits high strength with large tensile ductility (48% uniform elongation). Deformation twinning is strongly suppressed by the grain refinement, which is in good agreement with Shuhan et al. [31]. These findings reveal that superior ductility of TWIP steel is not only due to the deformation twinning but also to the inhibited dynamic recovery owing to the low SFE of the material [30].

Shuhan et al. [31] have studied an austenitic Fe-25Mn-3Al-3Si with varying grain size 7, 13, 30 and 63 μm. The strain hardening exponent increases when true strain ranges from 0 to 0.2, but becomes stable in the subsequent deformation. The coarse grain size stimulate the development of deformation twinning which enhance the work-hardening, on the other hand, fine grained specimen suppress the TWIP effect [31]. From research of Zhen-li Mi et al. [32], Fe-25Mn-3Al-3Si have been cold rolled with different strains with various annealing temperatures and results show that the cold rolled reduction of 65% and 1000 °C annealing temperature reveal the best
properties, yield strength of 225 MPa, tensile strength of 640 MPa, and the elongation at 82%. Mechanical twinning certainly makes a great contribution [32]. Another work done by Zhen-li Mi et al. [12], was conducted with different annealing temperature only. Two studies are in good agreement. In addition, with grain size between 20 and 40 μm, the deformation twinning reached its highest kinetics which resulted in excellent ductility [12]. Dini et al. [33] have investigated the Fe-31Mn-3Al-3Si TWIP steel with various grain sizes. With larger grain size, the onset of twins shifts to lower strain. With increasing strain, the presence of twinning increases resulting in the increase of work hardening, and the ductility is enhanced.

Meng et al. [34] and Yang et al. [35] have studied a high manganese Fe-33Mn-3Al-3Si TWIP steel by the means of compression and tensile tests. EBSD was adopted to analyse the microstructure. Mechanical twinning can be observed in both tension and compression tests. However, during compression tests, EBSD exhibits that twinning is related to grain orientation and distinct grain rotations, which resulting a special microstructure and suppresses the kinetics of twinning while comparing with tension deformation [34, 35].
From A. Petein et al. [36], Fe-15.99Mn-3.08Al-2.80Si and Fe-19.66Mn-3.11Al-2.88Si were investigated with two different annealing temperatures, 900 °C and 1000 °C. The evolution of the work-hardening rate and the transformation rate were alike. The phase transformation rate dominates the mechanical properties of the material. More austenite to martensite transformation is observed in steel Mn20 than in steel Mn15 and it shows greater work-hardening rate. With increasing annealing temperature, the grain size becomes larger. Mechanical twinning was not found in neither steel Mn15 nor Mn20.

Da-zhao et al. [37] report a Fe-30Mn-3Al-3Si-0.6C undergoing compression tests. XRD and TEM were used to study the microstructure evolution. The results present that as strain and strain rate increase, the stress, microhardness and work hardening rate increase. Work hardening rate declines at some points due to adiabatic heating. Many pin-like mechanical twins appear after impact loading. The grain size increases after deformation because of re-crystallization. Dominant deformation mechanisms are the interaction of twins with twins and twins with dislocation, and the appearance of multiple mechanical twins. The twin formation is attributed to “rebound mechanism”. At low strain, immature twins were observed. With deformation proceeds, deformation twins combine together. Moreover, twins
become denser as strain rate increases. Sahu et al. [38] have investigated Fe-24Mn-1Al-0.5Si-0.1C and Fe-24Mn-3Al-0.5Si-0.1C by a series of compression tests, including split Hopkinson bar technique. XRD and EBSD were utilized for the purpose of microstructure analysis. Results revealed that Al addition stabilize the austenite. At lower strain rate, the phase transformation was observed. While at higher strain rate, no austenite to martensite transformation takes place because adiabatic heating causes dynamic recrystallization.

Another study, with a Fe-31Mn-3Al-3Si TWIP steel, Dini et al. [39] used XRD technique to measure the dislocation density. Results showed that that the flow stress is influenced by two factors, one is dislocation-dislocation interactions, and the other is dislocation-mechanical twin boundary interactions. An effective procedure to produce submicron grained Fe-31Mn-3Al-3Si TWIP steel was suggested by Dini et al. [40] also, that is, large cold roll reduction followed by annealing. Wang et al. [41] have studied a twin-roll strip casting Fe-23Mn-3Si-3Al TWIP steel by means of SEM and TEM. Severe edge cracks of hot rolled TWIP steel are excluded while fabricating the TWIP steel by twin-roll strip casting.
Bajor et al. [42] investigated a Fe-29Mn-3Al-3Si TWIP steel by compression test and drawing process. Finding derived that the TWIP steel has good performance in cold drawing process. Sabet et al. [43] have investigated a Fe-30Mn-2.4Al-0.3Si undergoing hot compression tests and the high temperature slow behaviour was analysed. Dobrzański et al. [44-46] have conducted a series of hot compression tests on Fe-27Mn-4Si-2Al-Nb-Ti and Fe-26Mn-3Si-3Al-Nb-Ti. From a compression test of Yang Ping et al. [47], axiotaxy of two high manganese steels, Fe-22Mn-2Al-3Si and Fe-18Mn-2Al-3Si was studied.

To summarize above researches regarding the high Mn Fe-Mn-Al-Si system steels, with increasing manganese Mn contain, which increases the SFE, the mechanical twinning is likely to occur during deformation. In contrary, martensite transformation happens in low Mn steels, and as deformation proceeds, mechanical twins generate and increase, simultaneously, deformation mechanism changes from phase transformation to mechanical twinning, resulting in work hardening and enhance the strength. The ductility is improved as well. Twinning increases the plasticity of the materials while TRIP effect enhances strength of the materials. With increasing manganese content, the strength decreased, while the plasticity of the material increased. This indicates that TRIP or TWIP effect plays the
major role to the strength of these steels rather than solid solution strengthening. The finer grain size suppresses mechanical twinning. Alloys with larger grain size allow twins form easily. Increasing annealing time enlarges the grain size. Less twinning is observed during compression tests in comparison with tensile ones.

2.1.3 Fe-Mn-C system

ARCELOR and TKS have developed a fully austenitic carbon steels, namely X-IP, consisting of 17–24% of manganese and 0.5–0.7% of carbon. This Fe-Mn-C type material performs both mechanical twinning and ε-martensite formation in addition to dislocation slip due to its low SFE. This novel material has a tensile strength greater than 1000 MPa for a total elongation superior to 50% [48, 49]. The mechanical properties, formable grades, spot welding parameters, crash resistance were investigated and the impact resistance is of important in particularly. This high energy absorption characteristic can be defined as dissipation energy per unit volume. The value obtained from this specific Fe-Mn-C steel is 0.50 J/mm$^3$ while other conventional deep drawing steels only reach about 0.20-0.22 J/mm$^3$. The exceptional mechanical characteristics of this product are perfectly adapted to innovative steel design solutions for automotive body.
Barbier et al. [50] have investigated the microstructure and texture evolution of a fine grained Fe-22Mn-0.6C and reported that there is a weak decrease in strain hardening which is probably resulted from the accumulated dislocation pile-ups that form sub-boundaries impeding further twinning. Actually, the grain size has been decreased due to the formation of twins. Therefore, higher stress is needed to generate new twins as deformation proceeds. Mi et al. [12] used an in situ TEM to study a tensile Fe-23Mn-0.6C. Other than the basic deformation mechanism, dislocation gliding, mechanical twinning was also observed. Grain size was decreased by twin boundaries. The distance of twins reduced as stress increases and twins are prolonged. During fracture, micro-cracks originated from inclusions and twin-twin intersections. Bracke et al. [13] have adopted TEM and EBSD on a cold rolled Fe-22Mn (with minor C and N) TWIP steel to identify the main deformation mechanisms which were found to be micro-twinning and slip. Shear bands were also observed. The obtained crystallographic texture is brass-type, which is typical for low-SFE alloys. Twinning definitely has great influence in this brass texture developing. In addition, slip also plays an important role [13]. In a work done by Gutierrez-Urrutia et al. a novel SEM-based Electron Channelling Contrast Imaging (ECCI) technique was utilized to capture the dislocation cells and mechanical twins of a Fe-22Mn-0.6C steel [51]. There is a
strong interaction between dislocation slips and mechanical twinning. Bouaziz et al. [52] have investigated a pre-strain Fe-22Mn-0.6C. Twinning was found at the subsequent tensile test. Gutierrez-Urrutia et al. [14] reported that grain refinement does not suppress twinning with the tensile deformed Fe-22-Mn-0.6C TWIP steel at room temperature. The onset of mechanical twinning was observed while yielding with both materials with different grain sizes of 3 μm and 50 μm. Park et al. [53] have studied the tensile deformed microstructure of a Fe-22Mn-0.6C-xAl (x=0, 3, and 6) in detail.

A.S. Hamada et al. have conducted hot compression tested on a Fe-22Mn-0.14C followed by different treatments. Air cooling or water quenching resulted in fine grain size, around 10 μm, which suppressed the austenite to martensite transformation [54]. Wolfgang Bleck et al. [55] examined the hot workability of three different high Mn steel, Fe-23Mn-0.6C, Fe-16Mn-0.8C, and Fe-9Mn-0.9C. Mujica et al. [56] have investigated the laser-welded joints of Fe-22Mn-0.6C steel. Gutierrez-Urrutia et al. [57] have conducted a Bauschinger-type test (shear test) on Fe-22Mn-0.6C. Chen et al. [58] have studied the Portevin-Le Châtelier effect of Fe-18Mn-0.6C. This specific effect of Fe-20Mn-1.2C was also researched by Renard et al. [59].
In short, Fe-Mn-C system, during deformation, there is a strong interaction between dislocation gliding and mechanical twinning. The onset of mechanical twinning was observed while yielding Dislocation gliding is the main mechanism during deformation while twinning is dominant with carbon free high Mn steel. Grain refinement does not suppress twinning. Grain size decreases due to twin boundaries. The distance of twins reduced as stress increases and twins are prolonged. During fracture, micro-cracks originated from inclusions and twin-twin intersections.

2.1.4 Fe-Mn-C-Al system

Another group of high strength light weight steels is characterized as TRIPLEX, having decreased density. The chemical composition of these steels is Fe-26/30Mn-10/12Al-0.9/1.1C. Shear band Induced Plasticity (SIP-effect) is the main deformation mechanism which results in the superior ductility. Due to the reduced density (10–12% less), high strength level (>1000 MPa), beneficial formability including the high resistance to dynamic loading, the TRIPLEX alloys are of great perspective on the wide application in automotive industry, cryogenic technique [26, 60].

From an investigation of Je Doo Yoo et al. a Fe-28Mn-9Al-0.8C steel, increasing grain size (5, 8, 38 μm) results in increasing elongation (71%, 82%,
100% total elongation) while strength decreases (955, 903, 843 UTS, MPa) [61]. Another report of Je Doo Yoo et al. [62] shows neither transformation induced nor twinning induced plasticity happen in the fully austenitic Fe-28Mn-9Al-0.8C steel due to high Al addition resulting in austenite stabilization and high stacking fault energy. Dislocation cell formation was restricted as well. Microband induced plasticity is the main deformation mechanism [62]. From the beginning of plastic deformation to the medium strain level, in fine grain steel, the strain hardening rate stay unchanged. On the other hand, the strain hardening rate of coarse-grained steel keeps increasing to the high strain level, resulting in notable superior ductility. This is related to the formation and intersection of microbands, namely microband-induced plasticity. Another report addresses that Fe-28Mn-10Al-1.0C also shows extraordinary high uniform elongations (85-100%) and total elongations (100-110%) at room temperature [63].

To sum up, Fe-18/30Mn-9/12Al-0.7/1.2C steels exhibit significant weight reduction and superior mechanical, total elongation (>60%) and strength (700-1100MPa). Increasing grain size decreases strength while improves ductility. The main deformation mechanism is shear band induced plasticity or microband induced plasticity.
2.1.5 Fe-18Mn-0.6C-1.5Al (current research)

Jin et al. (2009) have examined a Fe-18Mn-0.6C-1.5Al in tensile test with $1 \times 10^4 \text{S}^{-1}$ strain rate. The strain hardening behaviour was analysed in detail, and the true tensile stress-strain curve and strain hardening rate are shown in Figure 2-8.

![True tensile stress-strain and strain hardening rate of Fe-18Mn-0.6C-1.5Al.](image)

As shown, the yield strength and the ultimate tensile strength is 317 and 1393 MPa, respectively. The strain hardening rate is high and the amount of strain hardening is more than 1000 MPa, which are principally due to the mechanical twinning since no strain-induced martensite was observed in the
fractured specimen. The plastic deformation is divided into four sub-regions. The various growth directions of mechanical twinning are generated while strain is more than 0.21 and the rate of twinning is significantly decreased when strain is greater than 0.37, which discloses that the strain hardening rate is positive proportional to the rate of mechanical twinning. As deformation proceeds, the volume fraction of twinning increases due to the increasing in number rather than the lateral growth of individual twin.

From a study of Kim et al. [25], and they conducted an elaborate analysis of the Dynamic Strain Aging (DSA) and Static Strain Aging (SSA) of a tensile tested Fe-18Mn-1.5Al-0.6C with $1 \times 10^{-3}$ S$^{-1}$ strain rate by means of a novel techniques, in-situ strain analysis and high sensitivity infrared thermal imaging. The strain rate jump test was conducted also to measure the strain rate sensitivity [25]. Another study of Zavattieri et al. investigated the Portevin-Le Chatelier (PLC) effect in a Fe-18Mn-0.6C-1.5Al with a Digital Image Correlation (DIC) method [64]. Kang et al. reported tensile tested, $1 \times 10^{-2}$ S$^{-1}$ strain rate, Fe-18Mn-0.6C-1.5Al TWIP steels which are firstly 60% cold rolled and followed by varying annealing temperature, 550-1100 °C for 10 min [65]. In whole, with increasing annealing temperature, the tensile strength decreased and ductility was improved. However, there is a reversion between 700 and 800 °C owing to the carbide precipitation.
Furthermore, the Hydrogen Delayed Fracture (HDF) properties and internal hydrogen behaviour of a Fe-18Mn-0.6C-1.5Al was probed by means of slow strain rate test and thermal desorption analysis [66].

As for formability is concerned, Chung et al. have examined Fe-18Mn-0.6C-1.5Al steels in comparison with DP600, by a series of tests including simple tension test, simple compression test, hemispherical dome test, notch test, disk compression test, three point bending test, cylindrical cup drawing test [67]. TWIP steel shows poorer formability unlike typical ductile sheets. Moreover, Chen et al. (2010) also investigated the stretch-flangeability of Fe-18Mn-0.6C-1.5Al by means of hole expansion test with infrared thermography. While compared with a Ti interstitial-free (IF) steel, this TWIP steel shows poorer hole expansion properties [68].

2.1.6 Corrosion properties and fatigue behaviour

M. Bobby Kannan et al. [69] and M. Opiela et al. [70] have researched the corrosion properties and behaviour of Fe-29.5Mn-3.1Al-1.4Si and Fe-24.4Mn-1.6Al-3.5Si. Both TWIP steels show poor acid and chloride corrosion resistance comparing with IF steels, though this is less substantial in chloride solution. However, in alkaline solution, the TWIP steels exhibited no significant difference in corrosion resistance while comparing with IF
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steels [69]. The chemical composition is the main factor influencing the corrosion property which is related to the high dissolution rate of Mn and Fe atoms in chloride solution [70, 71]. According to A.S. Hamada et al. (2006), addition of Cr in Fe-23Mn-8.4Al-0.5Si-0.2C and Fe-24Mn-5.7Al-0.5Si-0.2C TWIP steels tends to increase the acid corrosion resistance [71].

The fatigue behaviour of Fe-22M-0.6C, Fe-22M-3Al-3Si, Fe-18Mn-0.6C, and Fe-16Mn-0.3C-1.5Al was investigated by A.S. Hamada et al. [72] and Niendorf et al. [73]. The ultrafine-grained, 1.8 μm, Fe-22M-0.6C was studied as well by the same group, A.S. Hamada et al. [74]. The results proposed that no twins are formed under cyclic loading. Niendorf et al. [75] reported a fatigue crack growth of Fe-22Mn-0.6C steel. No substantial twinning was observed in the cyclic plastic zone while it was present during the unstable crack growth.

2.2 Alloying concept of TWIP steel

2.2.1 Manganese

As the main alloying element in TWIP steels, manganese is an austenite stabiliser [76]. The basic function of manganese is controlling the stacking fault energy. Lee and Choi [77] compared their work with other
investigations as shown in Figure 2-9 which represents that with an increasing Mn content, SFE first declines to a minimum value and then increases. The deformation mode changes from the TRIP to TWIP with increasing Mn content, owing to the increase in SFE with the Mn content from low values (< 20 mJ/m²) to moderate values (> 20 mJ/m²), as tabulated in Table 2-1. Manganese has only a small effect on solid solution strengthening in high manganese steels but a strong effect on the deformation mechanism. In addition, the Mn content slightly increase the flow resistance (about 2 MPa/wt%) and with increasing Mn content, the $\gamma \rightarrow \epsilon$ transformation temperature is decreased [77].
Figure 2-9 Variation of SFE as a function of Mn content in Fe-Mn alloys [77].

Table 2-1 Typical composition of TRIP / TWIP steel

<table>
<thead>
<tr>
<th>Mn wt.%</th>
<th>Si wt.%</th>
<th>Al wt.%</th>
<th>Fe wt.%</th>
<th>Steel category</th>
</tr>
</thead>
<tbody>
<tr>
<td>15.8</td>
<td>3.3</td>
<td>2.9</td>
<td>balance</td>
<td>TRIP</td>
</tr>
<tr>
<td>20.1</td>
<td>2.8</td>
<td>2.9</td>
<td>balance</td>
<td>TWIP/TRIP</td>
</tr>
<tr>
<td>26.5</td>
<td>3.0</td>
<td>2.8</td>
<td>balance</td>
<td>TWIP</td>
</tr>
<tr>
<td>29.2</td>
<td>3.0</td>
<td>2.8</td>
<td>balance</td>
<td>TWIP</td>
</tr>
<tr>
<td>33.0</td>
<td>3.0</td>
<td>2.9</td>
<td>balance</td>
<td>TWIP</td>
</tr>
</tbody>
</table>

2.2.2 Aluminium

Al is solely a ferrite stabiliser, but it is also an element that rises the Ms temperature of steels. However, the addition of aluminium to high
manganese TWIP steels makes contributions in several aspects. Most importantly, aluminium is also an austenite stabiliser, which decreases the stacking fault formation probability [16, 78] and increases the SFE. Therefore, the austenite to martensite transformation is suppressed [79] as shown in Figure 2-10. Moreover, solid solution strengthening is achieved [80]. Finally, the corrosion resistance of the steels is improved due to the high passivity of aluminium. As the function of controlling SFE, Dumny et al. [81] and Oh et al. [23] report that the SFE rises ~5 mJ/m² with 1% Aluminium added in Fe-22Mn-0.6C steel, ~10 mJ/m² with 1% Aluminium added in Fe-19Mn-5Cr-0.5Al-0.25C alloy. Tian et al. [82] have investigated an austenitic Fe-25Mn-(1.16~9.77)Al-0.68C (at%) alloys and discovered that SFE linearly increases with Al < 6.27 at%, and significantly with Al > 6.27 at%. Consequently, Al minimizes the Mn content. Al alloying in high Mn steels significantly suppresses the ε-martensite formation. Only by adding 0.5%Al, γ→ε transformation is suppressed for the Fe-27 %Mn steel, while 2.0%Al addition is required for the Fe-17 %Mn steel [83, 84]. The addition of aluminium to high Mn steels remarkably decreases the γ→ε transformation temperature [85]. Al addition can also increase the hot deformation resistance (about 12 to 15 MPa/wt% up to 6 wt%Al) [21, 86] (with Al alloying
up to 6%). For Al $\geq$ 6%, the high temperature promotes the ferrite phase formation, decreasing the flow stress [87].

![Figure 2-10 Al content vs. volume fraction of $\varepsilon$-martensite in high Mn steels [27].](image)

2.2.3 Silicon and carbon

The SFE of a high Mn steels is decreased by adding silicon which sustains the $\gamma$-$\varepsilon$ transformation during cooling and deformation [83, 88]. It was reported that with addition of 2% Si to Fe-27Mn steel, SFE decreases, as a result, the number of stacking faults increasing, which refine $\varepsilon$-martensite plates and increase fracture strength, but ductility is deteriorated [83]. Likewise, addition of Si strengthens the alloy as a result of the solid solution hardening [80]. It is generally acknowledged that carbon has high level of solubility in
austenite. Therefore, carbon is considered an effective austenite stabilizer and it strengthens the steel by solid solution hardening.

2.2.4 Other alloying elements

Chromium (Cr) content decreases stacking fault energy in the high manganese Fe-Mn-C system alloy [81]. However, the SFE is raised by adding Cr to a Fe-30Mn-6Si alloy [89]. Furthermore, corrosion resistance is enhanced by Cr addition [71], which is related to the chemical composition rather than phase structure. Huang et al. [90] have measured the stacking fault probability and observed that the Niobium (Nb) addition increased the stacking fault energy, retarded the martensite transformation, which decreased the tensile strength and increases the elongation of the TWIP steel [90]. The addition of nitrogen and the combination of Nitrogen (N) and Cr content in TWIP steel decreases the stacking fault probability and increase the SFE [78, 91]. N addition increases the ductility and a favourable strain-hardening behaviour.
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Table 2-2 is to summarize the effect of each individual alloying element in high manganese steel.

Table 2-2 Alloying concept of high Mn Steels. +: increasing effect, -: reducing effect.

<table>
<thead>
<tr>
<th>Element</th>
<th>Austenite stabiliser</th>
<th>Stacking fault energy</th>
<th>Solid solution strengthening austenite</th>
<th>Martensite formation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mn</td>
<td>+</td>
<td>- → +</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>+</td>
<td>+</td>
<td>+</td>
<td>-</td>
</tr>
<tr>
<td>Si</td>
<td>-</td>
<td>+</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>C</td>
<td>+</td>
<td>+</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>-</td>
<td>+</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nb</td>
<td>+</td>
<td>+</td>
<td></td>
<td>-</td>
</tr>
<tr>
<td>N</td>
<td>+</td>
<td>+</td>
<td></td>
<td>-</td>
</tr>
</tbody>
</table>

2.3 Evolution and formation of twinning

Jiménez et al. [92] reported that with interrupted tensile test on a Fe-22Mn-0.6C, there is a competition between mechanical twinning and dislocation gliding, which is in good agreement with Mi et al. [12], Fe-23Mn-0.6C, Rong-gang et al. [17], Fe-14,25Mn-3Al-2Si. The density of
dislocations and twins rapidly increases as strain increases. Lü et al. [93] have investigated a cold rolled Fe-22-0.367C steel and the microstructure observation showed that dislocation slip, twinning, ε-martensite formation and shear bending (at higher rolling strain) as deformation mechanisms.

From the research of Rong-gang et al. [17], Fe-14,25Mn-3Al-2Si, there are two steps sequentially occurring during twinning process. First, at a given stress, the onset of twinning in largest grain moves from the core to the next strong obstructions, such as grain or twin boundaries. Second, with applied stress increasing, the twin thickens and it’s linearly depends on the applied shear stress. Within individual grain, mechanical twinning and dislocation gliding are closely interacting and competing. Grain boundaries and twin act as strong barriers for twins [17]. Mi et al. [12] used an in situ TEM to study a tensile Fe-23Mn-0.6C. Other than the basic deformation mechanism, dislocation gliding, mechanical twinning was also observed. Grain size was decreased by twin boundaries. The distance of twins reduced as stress increases and twins are prolonged. During fracture, micro-cracks originated from inclusions and twin-twin intersections. R. Ueji et al. [30] have investigated a tensile tested Fe-31Mn-3Al-3Si with various grain sizes. The results showed that deformation twinning is strongly suppressed by the grain refinement, which is in good agreement with Shuhan et al. [31],
25Mn-3Al-3Si. While Gutierrez-Urrutia et al. [14] reported that grain refinement does not suppress twinning with the tensile deformed Fe-22-Mn-0.6C TWIP steel at room temperature.

As for the twinning nucleation process, from recent studies, H. Idrissi et al. [29] found that, with Fe-19.7Mn-3.1Al-2.9Si, at the beginning of plastic deformation, stacking faults appears moving from extrinsic to intrinsic. The deformation mode, simultaneously, alters from epsilon martensite formation to mechanical twinning [29]. Zhang et al. [28] reported that planar and wavy dislocations are observed at the onset of plastic deformation in Fe-30Mn-3Si-3Al. As deformation proceeds, mechanical twinning forms and increases. Similarly, Dini et al. [33] suggested that the pile-ups of planar dislocations have to be present before the twinning formation in Fe-31Mn-3Al-3Si. In addition, Park et al. [53] found that plastic deformation was activated planar glide of dislocations prior to mechanical twinning in Fe-22Mn-xAl-0.6C (x=0, 3, and 6). However, according to Gutierrez-Urrutia et al. [51] the mechanical twinning was firstly observed while yielding during a tensile deformation of Fe-22Mn-0.6C. Moreover, T.A. Lebedkina et al. [94] reckoned that twinning is the only cause of the plastic instability.
H. Idrissi et al. [8] also reported that the formation of twinning can be attributed to the pole mechanism. At the beginning of deformation of Fe-20Mn-1.2C, high level of sessile Frank dislocations are shown within mechanical twins. Da-zhao et al. [37] studied Fe-30Mn-3Al-3Si-0.6C undergoing compression test. The twin formation is attributed to “rebound mechanism”. At low strain, immature twins were observed, with deformation proceeds, deformation twins combine together. Moreover, twins become denser as strain rate increases.

It is generally accepted that various possible deformation modes, like twinning, dislocation gliding, ε-martensite formation can be closely interacting and competing. Twins form in the core of large grains and move to next strong obstacles, such as grain or twin boundaries. Twins become thicken, closer and longer as applied stress increases. The generation of twins decrease grain size. Consequently, the formation of new twins requires higher stress. As strain increases, the density of twins and dislocations increase rapidly. During the course of deformation, dislocation gliding plays the major role while twinning does not make significant contribution to overall texture, which is general observed by several investigations[6, 12, 92, 93]. D. Barbier et al. [50] also support this contention by the estimation of twin volume fraction (9%) of a Fe-22Mn-0.6C. However, Bracke et al. [13]
argued that mechanical twinning is the dominant deformation mechanism in a cold rolled Fe-22Mn. Moreover, in Fe-Mn-Al-Si steels, grain refinement suppresses deformation twinning while it not observed in Fe-Mn-C alloys.

2.4 Theoretical Approach

Physical-based models have been developed in order to analyse the interrelationship among deformation twinning mechanisms, compute the SFE by chemical composition, and predict the work-hardening owing to micro-structural refinement. Olivier Bouaziz’s group has been devoted into this area intensively [9, 11, 24, 95-97]. A physical based work hardening model of TWIP steel behaviour was proposed to describe the interaction between deformation twinning and dislocation gliding. The predictions from the model are in good agreement with experimental results, which verifies that twining acts as obstacles in dislocation motion [11]. Another verified model is to explain the isotropic and kinematic hardening in relation to the grain size and the twin spacing during straining [9]. Another model is, at different temperature, developed for the assessment of the stacking fault energy. The result exhibits that the plasticity mechanisms depending on the SFE; for SFE below 18 mJ/m², the mechanical martensitic transformation takes place while for SFE between 12 and 35 mJ/m², mechanical twinning
occurs [24]. A thermochemical model of the SFE with addition of Cu, Cr, Al and Si was presented. Aluminium significantly increases the SFE, while chromium reduces it. Copper also increases the SFE while silicon has more complex effect [97]. The formation of twins was proposed in a 2D simulation [95]. The model is capable of predicting several features of the microstructure such as the twin thickness and the stress field around twins.

In order to predict the mechanical properties of TRIP/TWIP steels, an Artificial Neural Network (ANN) model was developed. The input parameters are the contents of Mn (15~30wt%), Si (2~4wt%), and Al (2~4wt%), and the responses are the total elongation, yield strength and tensile strength. The experimental results are in satisfying agreement with the predicted data by ANN. With increasing manganese content, strength decreases while the elongation to fracture of the material increases, which indicate that TRIP or TWIP effects play the major role in strengthening of these steels other than solid solution strengthening [98]. A probabilistic model of strain hardening of Fe-Mn-based austenitic steel is presented and the predicted results exhibit good agreement with the published data [99].
2.5 Motivation of present work

The aim of current work was to fill in the gap of this particular Fe-18Mn-1.5Al-0.6C TWIP steel in literature which is lacking for the detail compression behaviour including low and high strain rates. This TWIP steel was also further investigated with an Explosive Bulge Test (EBT). The post-deformation microstructure and texture were characterized for better understanding of this TWIP steel.

The other objective of this work is to establish a CPFEM model of nano-indentation simulation in Abaqus environment to determine the unknown input parameters of the UMAT, which parameters can be of great importance in any other deformation simulation.
Chapter 3

Material and experimental instruments

The TWIP steel and experimental instruments and used in current study is included is this chapter.

3.1 Material used in current study

The material used for the present study is an austenitic Fe-18Mn-0.6C-1.5Al TWIP steel with a nominal ultimate tensile strength of 940 MPa. The chemical composition of this material is given in Table 3-1. The supplied as-received test material had been hot rolled from slab state and the most notable feature of the steel is the high level of manganese (18%) combined with significant levels of aluminum, carbon, chromium and Silicon.

Table 3-1 Chemical composition of the TWIP steel used in present investigation.

| Chemical Composition (in mass %) |
| Mn  | Al  | C   | Cr  | Si  | Ni  | Fe  |
| 17.85 | 1.31 | 0.592 | 0.368 | 0.223 | 0.101 | Bal. |
3.2  Mechanical testing instruments

3.2.1  Gleeble 3500 thermal and mechanical testing system

Gleeble 3500 is a fully integrated digital closed loop control thermomechanical testing system. Figure 3-1 gives a picture of the Gleeble 3500 facility located in the University of Wollongong. The thermal system is capable for heating up specimens at a rate of 10,000 °C/s, or maintaining a steady-state equilibrium temperature. With the high thermal conductivity grips, this facility is able to conduct a high cooling rate in excess of 10,000 °C/s at the surface of a specimen. On the other hand, the mechanical system is a complete hydraulic servo, and capable of practicing up to 10 tons of static force in tension or compression. The displacement rates can be reached to 1,000 mm/s. A series of low strain rates (1.0×10⁻²s⁻¹, 1.0×10¹s⁻¹, and 1.0×10²s⁻¹) of compression tests were performed on this facility.
3.2.2 Split Hopkinson pressure bar

A Split Hopkinson Pressure Bar (SHPB) is an apparatus for testing the dynamic stress-strain response of materials. In current research, this SHPB tests were conducted for high strain rate \(10^3\text{s}^{-1}\) compression tests by adopting a 13mm SHPB. The test setup is shown in Figure 3-2. Three methods of data analyses could be used which including (a) incident and transmission pulse, (b) incident and reflection pulse, (c) reflection and transmission pulse, and (d) incident, reflection and transmission pulse. Theoretically, if the testing material is fixed, the identical stress-strain
relationships would be obtained with different method (a, b, c and d). The signal is strictly based on the principle of 'Transmitted pulse=Reflected pulse+ Incident pulse'. However, it can be found that the stress-strain curve is different with different data analyse method, which means the incident, reflected and transmitted pulse don’t fit for the principle. This is mainly due to the system errors during the data acquisition, such as background noise, sensitivity coefficient of strain gauge, the mounted strain gauge is not perfect and the pressure bar is not straight etc. The aim of different data analysis method is to try finding which error is bigger than the others, for example, for concrete material the reflected pulse is preferred. For the TWIP steel, because the strength of the TWIP steel is very high, there is not plastic deformation generated. The elastic phase of stress-strain relationship of SHPB tests is not reliable. Normally, only the plastic phase of material is analysed.
3.2.3 Explosive Bulge Test (EBT)

Figure 3-3 shows the standard EBT setup in which the die block with the test plate located on top of it was placed beneath the suspended charge. The distance from the die block to the bottom of the charge was 320 mm. The plate had holes drilled into opposing corners to enable clamping with a bolted shackle in order to stop the blasted plates impacting the ceiling of the blast chamber. A TWIP steel plate (760 mm x 760mm x 8.5 mm) was selected as a trial material in this case. The EBT was performed using charge weight
(PE4 high explosive) of 2.3 kg. The explosive charge was cylindrical and 160 mm in diameter.

Figure 3-3 The EBT configuration shows that the plate is held on top of an annular-shaped die block and the charge was suspended from the ceiling in a stocking. ‘B-B’ represents the touching annulus between a plate and the die block. The inside diameter of the annulus B-B is approximately 635 mm. The A-B and B-C distances are 140 mm and 89 mm, respectively.

3.2.4 IBIS/UMIS nano-indentation system

In 1984, the IBIS/UMIT was initially manufactured by CSIRO Division of Applied Physics in Sydney Australia. The main purpose of this instrument
was designed for the hardness measurement of thin films. Various indenters, including Berkovich, spher-conical, knoop, and cube corner indenters can be mounted into this system. A picture of the system in the tribology laboratory at the University of Wollongong is shown in Figure 3-4. The nano-indentation information is obtained from penetration on loading and from the elastic recovery on unloading, which method can characterise bulk materials, such as metals, ceramics, plastics, crystalline and amorphous materials including coatings and modified surface layers. The load vs. depth curves can be continuously recorded by this system at a resolution of 75 nN in load and 0.05 nm in depth with a wide load range between 100 µN to 500 mN.

Figure 3-4 IBIS/UMIS nano-indentation system.
3.2.5 Hardness measurement

The Vickers hardness was measured on either Leco M-400-H1 hardness tester or DuraScan 70 automatic hardness tester as shown in Figure 3-5 and 3-6. The values given hereafter are the average of 3 to 5 measurement readings.

Figure 3-5 Leco M-400-H1.
3.3 Metallurgy and crystallography testing instruments

3.3.1 Metallurgy

Prior to analysing the microstructure of the material by microscope, the samples need to go through several preparation procedures. The applied steps are described as follow. Before observing the surfaces either parallel or perpendicular to the rolling direction, the samples have to be cut in desired direction. In order to minimize any unwanted mechanical damage during cutting, the Struers Accutom-50 automated cutting machine is applied and a Struers high quality cut-off wheel, 50A13, (with water as coolant) is used.
The force, feed speed, blade rotation speed were set as low, 0.03 mm/s, and 3000 rpm, respectively. The samples were then mounted in Polyfast powder by Struers CitoPress-20. As for nano-indentation samples, mounting step was not feasible. Afterwards, the grinding and polishing procedures were conducted either on the Struers automatic polisher Tegrapol 21 for mounted samples or on the Struers Rotopol-1 for unmounted ones. Those steps are given in Table 3-2. Special care was needed with polishing manually on Struers Rotopol-1.

Table 3-2 Sample preparation steps

<table>
<thead>
<tr>
<th>Surface</th>
<th>Force, N</th>
<th>Time (min)</th>
<th>Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grinding</td>
<td>SiC500</td>
<td>25</td>
<td>3</td>
</tr>
<tr>
<td>Grinding</td>
<td>SiC800</td>
<td>25</td>
<td>3</td>
</tr>
<tr>
<td>Grinding</td>
<td>SiC1200</td>
<td>25</td>
<td>3</td>
</tr>
<tr>
<td>Polishing</td>
<td>6 Nap cloth</td>
<td>20</td>
<td>10</td>
</tr>
<tr>
<td>Polishing</td>
<td>1 Nap cloth</td>
<td>15</td>
<td>10</td>
</tr>
<tr>
<td>Polishing</td>
<td>0.25 MD-Chem cloth</td>
<td>15</td>
<td>5</td>
</tr>
</tbody>
</table>
Etching was used to reveal the different aspects of the microstructure while using optical microscope. Nital etchant was found to present good result in visualize the microstructure of the Fe-17Mn-0.5C TWIP steel. The composition of 2.5% nital etchant is 2.5ml Nitric acid and 97.5ml Ethanol. While etching with Nital, timing was critical factor and the 5 seconds reveal the best results.

3.3.2 X-ray diffractometer

An X-ray diffraction (XRD) utilizing a GBC MMA diffractometer with Cu-Kα radiation was used to identify the phase composition of the samples before and after compression deformation. The operating voltage and current of X-ray beam was set as 35 kV and 28.6 mA, respectively.

3.3.3 ID15B beamline

ID15B beamlines are dedicated to applications using very high energy x-ray radiation up to several hundred keV which is located at the European Synchrotron Radiation Facility (ESRF) [100, 101] by using penetrating
high-energy X-rays of 86.94 keV. A fast Pixium 4700 flat-panel detector [102] was used for the purpose of data acquisition at ~2 Hz frame rate.

3.3.4 Optical Microscope (OM)

The optimal microscope used in current research is Leica DMR OM as shown in Figure 3-7. An etching procedure is needed prior to the microstructure analysis.

Figure 3-7 Leica DRM OM.
3.3.5 Scanning electron microscope (SEM)

The Scanning Electron Microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals disclose information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. JEOL 6490 SEM (Figure 3-8) was employed to study the microstructure of the TWIP steel. And the crystallographic orientation was obtained from JEOL 7001F (Figure 3-9) Field Emission gun Scanning Electron Microscope (FEG-SEM) by Electron BackScatter Diffraction (EBSD) technique.
Chapter 3 – Material and experimental instruments

Figure 3-8 JEOL 6490.

Figure 3-9 JEOL 7001F.
Chapter 4

Compression behaviour on TWIP steel

This chapter presents that a series of compression experiments were conducted on a austenitic Fe-18Mn-0.6C-1.5Al TWIP steel at various strain rates (from $1.0 \times 10^{-2}$ to $6.4 \times 10^{3} \text{s}^{-1}$) and total strains (~15% and ~20%) with a Gleeble 3500 thermo-mechanical simulator and a Split Hopkinson Pressure Bar (SHPB) system. Under compressive deformation, results showed this alloy possessed excellent strain-hardening behaviour, attributed to the occurrence of mechanical twinning during deformation. The prevailing deformation mechanism was observed to be twinning, which was substantiated by microstructural analyses, as well as phase identification and evolution of crystallographic texture. We also investigate the effect of both different deformation mechanisms and grain orientation on mechanical twinning in this TWIP Steel using microstructure observations by Electron BackScatter Diffraction (EBSD). The microstructure study shows that, under the same amount of deformation, the grain size appears smaller and less twinning boundaries were disclosed in compression. The grain oriented to A type of $<1 1 1>$ fibre when subjecting to tensile loading, while in compression, the brass type component was mainly observed.
4.1 Introduction

The amount of twinning and its morphology influences the mechanical properties significantly. As deformation proceeds, twins nucleate and grow in the core of large grains and move to the next strong obstacle, such as grain or twin boundaries. Subsequently, twins become thicker, closer spaced and longer as applied stress increases and it is believed that dislocation movement interacts and is hindered and obstructed by mechanical twinning [17]. In other words, as strain increases, the density of twins and dislocations increase rapidly. The grain size is reduced by the formation of twins (twin boundaries); consequently, higher stress is needed for new twins to form [4], which results in the rise of work hardening rate, enhancing the strength and increasing ductility [16].

It was reported [34] that during compression, the kinetics of twinning is suppressed in comparison with tensile deformation. When high Mn steels are deformed, the possible deformation mechanisms (e.g., dislocation slip, mechanical twinning and phase transformation) are closely interacting and competing with one another [12, 17, 29]. The potential of each mechanism is determined by the stacking fault energy (SFE), which is controlled by the chemical composition of the material [81]. The twinning effect is activated at
a low SFE (between 12 and 35 mJ/m²) [24] inherent in these materials. Grain size is a factor affecting twinning; in general, a coarse grain size (between 20-40μm) stimulates the development of deformation twinning which enhances the work-hardening, on the other hand, finer grain sizes suppress the TWIP effect [30, 33].

It is widely accepted that the uniaxial tensile deformation of FCC materials leads to a double fibre <1 1 1> and <1 0 0> texture components parallel to the loading direction [103]. The value of SFE of the materials influences the relative volume fraction of both fibres. For high SFE fcc materials, a dominant <1 1 1> fibre is displayed due to the ease of cross slip [103]. For intermediate to low SFE materials, the volume fraction of the <1 0 0> fibre rises with lower SFE, which is thought to be related to deformation twinning.

Texture evolution of high Mn TWIP steel has been investigated in studies involving behaviour due to cold rolling [13, 16, 104], as well as for tensile deformation [50]. The pronounced <1 1 1> fibre was reported prior to and after tensile deformation accompanied with <1 0 0> fibre, while the brass-type {1 1 0}<1 1 2> texture component was observed to be dominant during cold rolling, typical for low SFE alloys.
Recently, attention has been paid to the Fe-18Mn-0.6C-1.5Al, also referred to as TWIP940 [25, 67]. This particular alloy possesses excellent mechanical properties, as well as good energy absorption ability. However, literature is lacking for compression experiments and subsequent characterisations of the behaviour, microstructure, crystallographic analysis and deformation mechanisms of this alloy. Therefore, in this chapter, compressive testing (along with a tensile test) was undertaken at both low- and high-strain rates, in order to characterise mechanical property effects, as well as the evolution of microstructure and crystallographic texture, as a function of various strain rates and total strains.

4.2 Experimental

The nominal composition of the TWIP is detailed in chapter 3.1 with a final thickness of 2.24 mm, with average grain size of approximately 20 μm. The test specimens were cut to cylindrical shape of 5 mm in diameter for high strain rate tests and rectangular shape of 4×5×2.24 mm³ for lower strain rate tests. The reason for different specimen shape is due to the feasibility of the testing apparatus. The samples were compression tested at room temperature to two different total strains (~15% and ~20%) with the loading applied to the normal direction of the steel sheet. For the low strain rate
compression tests, at strain rates of $1.0 \times 10^{-2} \text{s}^{-1}$, $1.0 \times 10^{1} \text{s}^{-1}$, and $1.0 \times 10^{2} \text{s}^{-1}$, testing was performed using a Gleeble 3500 thermo-mechanical simulator, shown in chapter 3.2.1. For higher strain rate tests of $5.4 \times 10^{3} \text{s}^{-1}$ and $6.4 \times 10^{3} \text{s}^{-1}$, a SHPB system, detailed in chapter 3.2.2, was used. Microhardness was also undertaken using a Leco M-400-H1, shown in chapter 3.2.5, hardness tester with a 0.5 kg load, to determine the hardness effect from different strain rates and total strains.

After experiments, all specimens were mechanically ground and polished in accordance with standard procedures for steels and etched with 2.5% Nital, in order to reveal microstructures, which were subsequently observed with both a Leica DMR optical microscope (chapter 3.3.4) and a JOEL JSM-6490 (chapter 3.3.5) Scanning Electron Microscope (SEM). Phase identification of the test material before and after experiments were determined with a GBC-MMM X-Ray Diffractometer (XRD) shown in chapter 3.3.2. For the investigation of crystallographic texture, experiments were conducted at the ID15B beamline, as described in chapter 3.3.3. Crystallographic texture measurements were performed on as-received and ~20% total strain compressed specimens at various strain rates.
All detailed microstructural analyses were undertaken by examining the Transverse Direction (TD) of the specimens. Electron BackScattered Diffraction (EBSD) was conducted using a JEOL JSM-7001F (shown in chapter 3.3.5) Field-Emission Gun Scanning Electron Microscope (FEGSEM) fitted with a Nordlys-II (S) camera and AZtecHKL software. All EBSD was carried out at 15kV, with a step size of 0.5 µm and a total scanned area of 300 x 230 µm².

EBSD maps were imported into HKL Channel-5 software for the post-processing of the data obtained. For the band contrast maps, the black lines indicated High Angle Grain Boundaries (HAGBs) with critical misorientation > 15°; grey lines represented the Low Angle Grain Boundaries (LAGBs), 2° ≤ θ < 15° and misorientations less than 2° were disregarded. The Total High Angle Grain Boundaries (THAGBs) consisted of HAGB and Twin Boundaries (TBs). First order TBs were defined as Σ3=60° <111> and depicted by red lines; whilst second order TBs were Σ9=38.9° <101> and represented by blue lines. A tolerance limit of 6° for Σ3 and 2.4° for Σ9, was used following the Palumbo–Aust criterion [105]. The texture intensities \( f_r(g)\) along the <1 0 0> and \( a \) fibres were plotted along their ideal skeleton lines and the intensities along the <1 1 1> fibre were determined in Matlab using a 10° deviation limit from the ideal orientations.
4.3 Results and discussion

4.3.1 Stress-strain curves and mechanical properties

The true stress-strain curves obtained from both Gleeble 3500 and SHPB compression experiments are shown in Figure 4-1. Figure 4-1(a) shows the true stress-strain curves of ~15% total strain at various strain rate ranging from $1.0 \times 10^2 \text{s}^{-1}$, $1.0 \times 10^1 \text{s}^{-1}$, $1.0 \times 10^2 \text{s}^{-1}$ and $5.4 \times 10^3 \text{s}^{-1}$; while Figure 4-1(b) presents those curves of ~20% total strain at strain rates of $1.0 \times 10^2 \text{s}^{-1}$, $1.0 \times 10^1 \text{s}^{-1}$, $1.0 \times 10^2 \text{s}^{-1}$ and $6.4 \times 10^3 \text{s}^{-1}$. 
Figure 4-1 True stress-true strain plot of Fe-18Mn-0.6C-1.5Al at different strain rates for (a) ~15% and (b) ~20% total strain. Post-yield behaviour is shown.

In the current work, only the plastic behaviour of the compression true stress–strain curve is analysed, from yield point onward. This is because the tests undertaken using the Gleeble 3500 had slight irregularities within the elastic limit. Therefore, for consistency, the elastic portion was omitted in this work and these curves show only post-yield deformation behaviour for all tests. Table 4-1 shows the respective changes in yield stress and the work hardening rates, as a function of strain rate and total strain.
Table 4-1 Compressive properties of Fe-18Mn-0.6C-1.5Al steel at various strain rates with different total strains.

<table>
<thead>
<tr>
<th>Strain Rate ($S^{-1}$)</th>
<th>Yielding Stress (Mpa)</th>
<th>Work hardening rate (MPa/Unit strain)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>~15% strain</td>
<td>~20% strain</td>
</tr>
<tr>
<td>6.4×10³</td>
<td>N.A.</td>
<td>708</td>
</tr>
<tr>
<td>5.4×10³</td>
<td>672</td>
<td>N.A.</td>
</tr>
<tr>
<td>1×10²</td>
<td>566</td>
<td>602</td>
</tr>
<tr>
<td>1×10¹</td>
<td>552</td>
<td>556</td>
</tr>
<tr>
<td>1×10⁻²</td>
<td>511</td>
<td>531</td>
</tr>
</tbody>
</table>

The yield strength was observed to rise with the increase in strain rate, especially for higher strain rate SHPB tests, which was in good agreement with previous studies [4, 104]. It should also be noted that the higher value of yield stress was found when the total strain increases from ~15% to ~20%. The increase in both strain rate and total strain was observed to increase the strength of the material due to the formation of twins, which was subsequently validated by the microstructural analyses and the texture evolution of the deformed specimens, as described in further sections. In addition, from Table 4-1 the values of work hardening rate are of
approximately the same order. However, this increased slightly when the total strain increased from ~15% to ~20%. This behaviour corresponded to previous reports [4, 12, 17], whereby the generated twins (twin boundaries) cut down the grain size; as a result, higher stress is needed to generate new twins as deformation proceeds. Furthermore, the specific energy absorption of Fe-18Mn-0.6C-1.5Al is approximately 0.54 J/mm$^3$, which corresponds to more than twice the value of those of several conventional deep-drawing steels [4], and mechanical twinning is believed to be the key factor.

The relationship between strain hardening exponent and true stress-strain is proposed by Hollomon law [106]:

$$\sigma = K \varepsilon^n$$  \hspace{1cm} (4 - 1)

Where $\sigma$ is true stress, $\varepsilon$ is true strain, $K$ is strain hardening coefficient, $n$ is strain hardening exponent. By applying natural logarithm, Equation (4.2) can be obtained:

$$\ln \sigma = n \ln \varepsilon + \ln K$$  \hspace{1cm} (4 - 2)

Since slight irregularities occurred within the elastic limit of tests undertaken at strain rates of $1.0 \times 10^{-2} \text{s}^{-1}$, $1.0 \times 10^{1} \text{s}^{-1}$, and $1.0 \times 10^{2} \text{s}^{-1}$, Figure 4-2 shows only the
compressive lnσ-lnε curves of the two SHPB tests at strain rates of 5.4×10³s⁻¹ and 6.4×10³s⁻¹. It can be deduced that \( n_s \) is the slope of lnσ-lnε curves. Moreover, based on the calculated strain hardening exponents, both curves can be categorised into three stages. Table 4-2 lists the value of \( n \) at different stages of the compressive lnσ-lnε curves in Figure 4-2.

Figure 4-2 lnσ-lnε plot of the Fe-18Mn-0.6C-1.5Al steel at 5.4×10³s⁻¹ and 6.4×10³s⁻¹ with ~15% and ~20% total strain.
Table 4-2 Strain hardening exponent, n, of Fe-18Mn-0.6C-1.5Al steel at two high strain rates.

<table>
<thead>
<tr>
<th>Strain Rate</th>
<th>Stage I</th>
<th>Stage II</th>
<th>Stage III</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.4×10³</td>
<td>0.29</td>
<td>0.06</td>
<td>0.72</td>
</tr>
<tr>
<td>5.4×10³</td>
<td>0.86</td>
<td>0.10</td>
<td>0.75</td>
</tr>
</tbody>
</table>

During the deforming process of high Mn TWIP steel, it is well known that the two major deformation mechanisms, dislocation slip and mechanical twinning, are strongly interacting and competing with each other. The \( \ln \sigma - \ln \varepsilon \) curves signify that the onset of Stage II is also where plastic deformation commences, which signifies the leading mechanisms of Stage II is dislocation slip [37, 38]. On the other hand, Stages I and III are the twinning strengthening sections where \( n \) is comparatively high and with greater application of stress applied, secondary new twins form at Stage III. Li et al [37] reported that there was a thermal softening stage, possessing relatively small or negative values of \( n \), before Stage III which was not observed in this present study. Sahu et al. [38] also reported the enlarged grain size after SHPB test was caused by strain softening by dynamic recovery and recrystallization. However, the results in our current work do not show similar findings. This may be attributed to the dissimilar
composition of the steel, as the strain rates tested were within the same range as this investigation; and/or the specimens used in the current work being substantially smaller than those used in other investigations.

The microhardness evolution of the material in the as-received state and after compressive deformation is shown in Figure 4-3. As compared to the as-received specimens, the microhardness values were found to increase significantly after compression testing, signifying considerable work-hardening. A total strain of ~20% resulted in higher values than those of ~15%, which can be interpreted from the pronounced strain-hardening behaviour of Fe-18Mn-0.6C-1.5Al steel. The microhardness values of the specimens tested to a total strain of ~20%, were observed to be on average 8% greater than that of the sample subjected to a total strain of 15%. However, as is observed in Figure 4-3, a plateau of hardness values occurred in the three measurements between 1.0×10^{-2}\text{ s}^{-1} and 1.0×10^{2}\text{ s}^{-1}, with only a slight increase shown in the tests conducted at the higher strain rates of 5.4×10^{3}\text{ s}^{-1} and 6.4×10^{5}\text{ s}^{-1}, which appeared to suggest a saturation of work hardening at these strain rates.
Chapter 4 – Compression behaviour on TWIP steel

4.3.2 Optical microscopic and SEM observation

Figure 4-4 (a) shows the microstructure of the as-received and undeformed Fe-18Mn-0.6C-1.5Al steel, in which a fully γ-austenite microstructure is evident. Figure 4-4 (b-e) show microstructures from compression tests deformed to ~15% total strain at various strain rates. In comparison with the as-received specimen, all the microstructures from the compression-tested specimens displayed significantly deformed grains elongated perpendicular to the applied stress, along with twinning-like features.
Figure 4-4 Optical micrographs of the (a) As-received and ~15% deformed at (b) $1.0 \times 10^2$ s$^{-1}$, (c) $1.0 \times 10^3$ s$^{-1}$, (d) $1.0 \times 10^2$ s$^{-1}$, (e) $5.4 \times 10^3$ s$^{-1}$ strain rate, respectively.
A SEM micrograph of a sample compression tested at $1.0 \times 10^2 \text{s}^{-1}$ strain rate and $\sim 15\%$ total strain is shown in Figure 4-5. Mechanical twinning can be clearly observed (arrows imply areas of twins). These features were observed in all specimens that were compression tested, regardless of strain rate or total strain.

![SEM micrograph](image)

Figure 4-5 SEM micrographs of a compressive deformed Fe-18Mn-0.6C-1.5Al steel. Arrows depict region of twins.

4.3.3 Phase identification

As stress-induced phase transformation is one deformation mechanism observed in high Mn TWIP steels, Figure 4-6 presents XRD profiles of diffracted intensity vs. scattering vector $Q (= 4\pi \sin\theta/\lambda)$ for all strain rates of testing at both total strains of $\sim 15\%$ (Figure 4-6(a)) and $\sim 20\%$ (Figure 4-6(b))
to examine the occurrence of any phase transformation as a result of compressive deformation. From Figure 4-6, it is observed that there was no phase transformation apparent during deformation, as only single austenite phase was determined both before and after compression experiments. Therefore, it is suggested that mechanical twinning is one of the major deformation mechanisms, as no transformation-induced phases were observed in this study.
4.3.4 ID15B beamline results

To study the development of crystallographic texture as a result of compressive deformation, pole figures were derived from high-energy X-ray
diffraction data. In the form of stereographic projections, a pole figure is a two dimensional graphical representation of the orientation distribution of crystallographic lattice planes in the material. Among them, (111) pole figures were selected and displayed representatively here. Figure 4-7 (a) shows the typical rolling texture components in FCC alloys [107] as reference, consisting of ideal orientation components, including Cube \{100\}<001>, Goss \{110\}<001>, Brass \{110\}<112>, Copper \{112\}<111>, and S \{123\}<634>. From the experiments undertaken in this investigation, Figure 4-7 (b) displays the (111) pole figure of the as-received Fe-18Mn-0.6C-1.5Al steel, while Figures 4-7 (c-f) exhibit those of compression tested samples with ~20% total strain at various strain rate of (c) 1.0×10^{-2}s^{-1}, (d) 1.0×10^{1}s^{-1}, (e) 1.0×10^{2}s^{-1}, (f) 6.4×10^{3}s^{-1}. It can be inferred that for the as-received material, the texture component was mainly cube type \{001\}<100> which possess greater Schmid’s factor of dislocation slip [33], while all other deformed specimens showed a dominant brass type \{110\}<112> component with minor Goss type \{110\}<001> having higher values of Schmid’s factor in twinning [104]. These results also correspond to several reported works investigating the microstructure and texture evolution during cold rolling of high Mn TWIP steel [13, 16]. Therefore, it
can be concluded that mechanical twinning definitely plays a significant role in the process of deformation and dislocation slip also participates.
Figure 4-7 (1 1 1) pole figures of the (a) ideal rolling texture for FCC, and (b) as-received, (c) $1.0 \times 10^2 \text{s}^{-1}$, (d) $1.0 \times 10^1 \text{s}^{-1}$, (e) $1.0 \times 10^2 \text{s}^{-1}$, (f) $6.4 \times 10^3 \text{s}^{-1}$ strain rates.
4.3.5 EBSD results

Figure 4-8 shows the EBSD grain boundaries maps of the original, post 20% tensile and both 15%, 20% compression at various strain rate (as denoted) specimens, the horizontal is the transverse direction. From observation, at the same level of final strain, 20%, the grain size appears larger in tensile tested sample than in compression one. From all the samples of compression tests, it is clearly that the grain size has a trend to decrease with the strain rate. In all the samples, the first order TBs, $\Sigma 3$ (red lines) are evenly distributed while the second order TBs, $\Sigma 9$ (blue lines) is barely observed. LAGBs were heavily generated in all the deformed specimens.
Figure 4-8 EBSD maps of (a) original, (b) 20% tensile, 15% compression at (c) $10^2$, (d) $10^3$, (e) $10^3$ strain rate and 20% at (f) $10^2$, (g) $10^3$, (h) $10^3$ strain rate.
Figure 4-9(a) shows the change in misorientation distribution with the specimens of original, tensile, two levels of compression test, and the curve of the theoretical random orientation. Regardless of the variance of the final strain and strain rate, all the curves of compression tested specimens reveal the identical pattern; therefore, only the curves of 10^2 s^{-1} strain rate are representatively shown in the Figure 4-9(a). There are two bumps detected in the curve of original sample, with the two illustrations of the misorientation axis distributions in the crystal coordinate system, the small peak around 39° indicates the second order (Σ9) twin boundaries and the big bump at 60° is related to the first order twin (Σ3) boundary. As for deformed specimens, there is no peak found near 39°; however, peaks were detected at ~60°. At the area of low misorientation angles, the curve of tensile test appears highest peak whereas the curve of original sample displays the lowest; while at ~60°, the highest bump was from original sample and the lowest was found from compression ones. The specific boundary area fraction can be calculated from the misorientation distribution and plotted as Figure 4-9(b). In the case of compression tests, since the factor of strain rate doesn’t have significant effect on the value grain boundary area fraction while the level of final strain does, the average value (along with the standard deviation error) of the boundary area fraction from three different strain rates was adopted here for each final
strain. The area fraction of HAGBs, $\Sigma 3$ TBs and $\Sigma 9$ TBs decrease after tensile test and those values drop even further with compression experiment which is related to the increase in LAGBs. Figure 4-9(c) illustrated the both first ($\Sigma 3$) and second ($\Sigma 9$) order TBs length fraction of all specimens as marked.

Figure 4-9 (a) Change in misorientation distribution as a function of various specimens, (b) grain boundary area fraction as a function of degree of strain, and (c) evolution of length fraction of $\Sigma 3$ and $\Sigma 9$ twin boundaries with various strain rate and final strain.
Figure 4-10 shows the relationship between crystallite grain size (with TBs and without, W/O TBs) and the HV hardness.

![Graph showing grain size and hardness](image)

Figure 4-10 (a) and (d) change in grain size with and without taking TBs into account with various specimens and (b) hardness vs grain size of various specimens with and without taking TBs into account.

The grain size refined after deformation and the larger strain rate resulted in finer grains, as seen from Figure 4-8 and Figure 4-10, which finding is against with the Li’s SHPB work on another TWIP steel [6, 37]. Li claimed an adiabatic process has occurred during the deformation which causes the dynamic recovery and recrystallization leading to increase in grain size after deformation. The reason to this disagreement might be due to the significant difference in the composition. The Mn content of TWIP940 is nearly half of the one used in Li’s work. The grain refinement after deformation obtained in current investigation results in the strengthening of the TWIP940 [108], the
increase of hardness. This is concluded that the main strengthening mechanism is work hardening effect, known as Hall-Petch strengthening.

From Figure 4-9(b), at the same level of deformation (20%), the area fraction of $\Sigma 3$ TBs obtained from tensile test is triple that from compression. It is also observed that less twinning activity occurred with higher compression strain (20%). It implies that the compression test suppresses the mechanical twinning compared with tensile test, which is in good agreement with Meng’s finding [34]. Attention should also be paid to the relatively lower fraction of $\Sigma 9$ TBs compared to $\Sigma 3$ TBs in Figure 4-9(b-c).

For the $\phi_2 = 0^\circ$, $45^\circ$ and $65^\circ$ Orientation Distribution Function (ODF) sections, the main texture components of fcc materials are schematically represented in Figure 4-11 and Table 4-3. The $<1\ 1\ 1>$, $<1\ 0\ 0>$ and $<1\ 1\ 0>$ fibres are indicated on the ODF sections with a spread of $15^\circ$ around the ideal skeleton lines. The evolution of textures with original and various deformation conditions are given for the $\phi_2 = 0^\circ$, $45^\circ$ and $65^\circ$ ODF sections in Figure 4-12. Along the various fibres, the change in the intensity ($f(g)$) of the individual texture components is plotted in Figure 4-13(a-c). The intensity of important texture components of pre-, and post-deformation are shown in Figure 4-13(d). The original sample is characterized with the maximum intensity of
3.5 and a texture index of 1.46 while at 20% tensile strain, the maximum intensity of 4.8 lies on the <1 1 1> fibre with 1.64 texture index. As for all other compressed specimens, the maximum intensity was found to be located at the brass component. The texture index is to evaluate the overall texture strength which is shown in Table 4-4. It is noted that the texture index increase with the further strain (from 15% to 20%) and the strain rate.

Figure 4-11 A diagram of the important texture texture components in fcc materials. <1 1 1> — red, <1 0 0> — blue, <1 1 0> — green.
Table 4-3 Euler angles and Miller indices for common texture components in fcc metals and alloys.

<table>
<thead>
<tr>
<th>Texture component</th>
<th>Symbol</th>
<th>Euler angles</th>
<th>Miller indices</th>
<th>Fibre</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cube (C)</td>
<td>45 0 45</td>
<td>[0 0 1]&lt;1 0 0&gt;</td>
<td>&lt;1 0 0&gt;</td>
<td></td>
</tr>
<tr>
<td>Goss (G)</td>
<td>90 90 45</td>
<td>[1 1 0]&lt;0 0 1&gt;</td>
<td>&lt;1 0 0&gt;</td>
<td></td>
</tr>
<tr>
<td>Brass (B)</td>
<td>55 90 45</td>
<td>[1 1 0]&lt;1 1 2&gt;</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>35 90 45</td>
<td>[1 1 0]&lt;1 1 1&gt;</td>
<td>&lt;1 1 1&gt;</td>
<td></td>
</tr>
<tr>
<td>Rotated Goss (Rt-G)</td>
<td>0 90 45</td>
<td>[0 1 1]&lt;0 1 1&gt;</td>
<td>&lt;1 1 0&gt;</td>
<td></td>
</tr>
<tr>
<td>Rotated cube (Rt-C)</td>
<td>0/90 0 45</td>
<td>[0 0 1]&lt;1 1 0&gt;</td>
<td>&lt;1 1 0&gt;</td>
<td></td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>90 35 45</td>
<td>[1 1 2]&lt;1 1 1&gt;</td>
<td>&lt;1 1 1&gt;</td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>59 37 63</td>
<td>[1 2 3]&lt;6 3 4&gt;</td>
<td>–</td>
<td></td>
</tr>
<tr>
<td>~S</td>
<td>75 37 63</td>
<td>[1 2 3]&lt;1 1 1&gt;</td>
<td>&lt;1 1 1&gt;</td>
<td></td>
</tr>
</tbody>
</table>
Figure 4-12 $\phi_2 = 0^\circ$, $45^\circ$ and $65^\circ$ ODF sections of the (a) original, (b) 20\% tensile samples, and compression experiments at 10-2 strain rate with (c) 15\%,
(d) 20% final strain, at 102 strain rate with (e) 15%, (f) 20% final strain, and at 103 strain rate with (g) 15%, (h) 20% final strain. Contour levels = 1×.
Figure 4-13 The variation of texture intensity (fe(g)) along (a) α, (b) <111> and (c) <100> fibres. (d) the intensity of important texture components with various specimens.

Table 4-4 The texture index of various specimens.

<table>
<thead>
<tr>
<th>Original strain rate</th>
<th>Tensile strain</th>
<th>Compression strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>10^{-4}</td>
<td>20%</td>
<td>15%</td>
</tr>
<tr>
<td>10^{-2}</td>
<td>20%</td>
<td>20%</td>
</tr>
<tr>
<td>10^{-2}</td>
<td>15%</td>
<td>20%</td>
</tr>
<tr>
<td>10^{2}</td>
<td>15%</td>
<td>20%</td>
</tr>
<tr>
<td>10^{2}</td>
<td>20%</td>
<td>15%</td>
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<tr>
<td>10^{3}</td>
<td>20%</td>
<td>20%</td>
</tr>
<tr>
<td>10^{3}</td>
<td>15%</td>
<td>20%</td>
</tr>
<tr>
<td>Texture index</td>
<td>1.46</td>
<td>1.64</td>
</tr>
<tr>
<td></td>
<td>1.49</td>
<td>1.73</td>
</tr>
<tr>
<td></td>
<td>1.62</td>
<td>1.92</td>
</tr>
<tr>
<td></td>
<td>1.76</td>
<td>2</td>
</tr>
</tbody>
</table>
The texture of the original TWIP940 steel exhibited mainly the cube type \{0 0 1\}<1 0 0>, the brass type \{1 1 0\}<1 1 2>, and the copper (Cu) \{1 1 2\}<1 1 1> components as seen in Figure 4-12(a), possessing a texture index of 1.46. Which result is quite matching with other works [5, 92] of high manganese (Fe-22Mn0.6C and Fe-24Mn-3Al-2Si-1Ni-0.06C (wt. %)) TWIP steel other than the texture is slightly stronger here. After tensile testing, the texture analysis, Figure 4-12(b), discloses the enhanced A\{1 1 0\}<1 1 1> type and weakened cube \{0 0 1\}<1 0 0>, copper \{1 1 2\}<1 1 1> and ~S \{1 2 3\}<1 1 1> orientation. These components belong either to the pronounced \<1 1 1>/TD fibre (A, copper and ~S) or to the weak \<1 0 0>/TD fibre (cube), which finding is quite corresponding to several other texture study works of high Mn TWIP steel [5, 50, 92] as well.

On the other hand, Figure 4-12(c-h) indicate dominant brass type component developed after each single compression test, which is in good agreement with a previous high-energy X-ray diffraction work [108]. An obvious increase (triple the value) in the intensity of the brass orientation is observed in the \(\alpha\) fibre plot (Figure 4-13(a)), whereas A orientation of the tensile sample appeared the highest value of the intensity (Figure 4-13(b)). Other than the brass type component, minor A, S, cube and copper type components accompanied as well. From Figure 4-13(c) and 4-13(d), the cube
type component revealed the decreased intensity; on the contrary, the brass, Goss and A orientation were enhanced. The Rotated-Goss [0 1 1]<0 1 1> of <1 1 0> fibre doesn’t show the constant pattern which isn’t in concord with the investigation of Meng [34]. The randomness of the minor texture component might be attributed to the relative small size of the sample and the ultra-high strain rate of the compression experiments leading to a tiny deviation of the EBSD orientation calibration.

4.4 Conclusions

Compression deformation experiments at a variety of strain rates and two total strains were undertaken on Fe-18Mn-1.5Al-0.6C steel and mechanical properties including strength and hardness, as well as microstructural evolution and crystallographic texture of the material were studied. The results indicate that the strength and hardness of the material were increased by either increasing strain rate or total strain and the promising combination of properties renders the outstanding ability of energy absorption. Mechanical twinning is well believed to be the cause of the superior strain hardening behaviour, which was jointly verified by microstructural characterisation, phase identification and texture analysis. That is, after deformation, the microstructure revealed the presence of twinning and no
phase transformation was observed. Accordingly, the activity of twinning was further supported by the results of high-energy x-ray diffraction experiments, revealing the texture evolved principally from cube type \{0 0 1\}<1 0 0> to brass type \{1 1 0\}<1 1 2> components after compressive deformation, therefore showing that the twinning heavily occurs during deformation of this steel, while slip also makes a contribution. After tensile experiments, the first order twinning boundary area fraction decrease from 31.4\% to 15.9\% (about half), following compression tests the value drop to 6\%. The results indicate that the compression suppress twinning activity comparing to tensile loading. The grain size decrease subsequent to deformation; compressed samples have smaller value of grain size to that of tensile one. A relatively stronger A type component of \langle1 1 1\rangle fibre texture was observed in the tensile sample while the brass type shows as dominant orientation in compressed specimens. The Goss type component intensified after deformation while the cube type weakened.
Chapter 5

Effects of dynamic impact loading on TWIP steel

The work described here is a microstructural investigation of TWIP steel that has been subjected to blast loading. It is found that the pre-blast technique reduced the grain size of the TWIP steel significantly. The reduction in grain size resulted in a considerable increase in material hardness.

5.1 Introduction

There is an ongoing demand to develop new materials for motor vehicles with improved crash survivability and reduced weight/maintenance. As far as armoured vehicles are concerned, which are primarily designed to provide protection against blast and ballistic events, and there is a pressure to reduce the weight of vehicles in order to achieve improvements in range and manoeuvrability combined with reductions in operating cost.

It happens that materials for armour applications require similar energy absorbing properties when the major threat is from blast loading. The key requirement of an innovative material is, under severe deformation, the capability to absorbing maximum energy without failing catastrophically. A
concept of Ultra-Fine Grain (UFG) was adopted in this chapter to look at the possible method to achieve the desired properties. From a fundamental viewpoint, as indicated by the Hall-Petch equation, UFG is an ideal means for hardening and strengthening a metal without changing its chemical composition and compromising ductility.

In steels, transformation strengthening (from martensite) is the major strengthening mechanism. Generally, there are five different types of strengthening mechanisms for metals: solid solution hardening, precipitation hardening, dispersion hardening, work hardening and grain refinement hardening. The adopted strengthening mechanism in this chapter was grain refinement hardening by reducing grain size through plastic deformation. UFG materials generally show some excellent properties such as ultrahigh strength, enhanced fatigue behaviour and superior corrosion resistance. If grain refinement strengthening is effective for auto-related applications it may be also suitable for armour applications, which require similar properties.

One efficient technique for producing UFG materials is Severe Plastic Deformation (SPD). Several SPD processing techniques have been developed to obtain UFG structures in both bulk and sheet materials. These techniques
include equal-channel angular pressing [109-111], high-pressure torsion [112], multi-axial compression/forging [113] and accumulative roll bonding [114, 115]. Most techniques for SPD use relatively static or quasi-static low strain rate techniques, whereas blast loading is a rapid straining technique and there is insufficient information concerning the effect of rapid/high impact loading on grain size and the consequent effect of this process on strength and ductility. Therefore it is worthwhile to study the effect on microstructure (especially on grain size) of materials subjected to blast loading.

It is clear from Equation 4-2, $\ln \sigma = n \ln \varepsilon + \ln K$, $n$ plays a dominant role over instantaneous hardening rate. The $n$ (work hardening exponent) value depends on the atomic structure, for Face Centre Cubic (FCC) $n < 0.6$, for Body Centre Cubic (BCC), $n < 0.2$ and for Hexagonal Close Packed (HCP), $n < 0.0002$. For this reason, it is desirable to select FCC structures to study the effects of UFG. Furthermore among FCC structures, the chosen material should have very high energy absorption ability to obtain improved performance without compromising blast and ballistic properties. High manganese TWinning Induced Plasticity (TWIP) steel is one of the best candidates for this work. This material also satisfies the requirements of toughness, ductility and corrosion resistance.
In this chapter, a plate of TWIP steel that has been subjected to standard Explosion Bulge Testing (EBT) is investigated to identify the effect of blast loading on material microstructure and hardness.

5.2 Experimental

The material used for the present study is an austenitic TWIP steel which was addressed in chapter 3.1. The standard EBT setup is detailed in chapter 3.2.3. In this investigation, the EBT was carried out at ambient temperature (≈17°C) and deformation resistance of the plate was measured in terms of bulge depth and plate thinning. The bulge depth of the plate was measured as a distance from the flat surface of the plate to the maximum bulge at the centre of the plate after a blast. Figure 5-1 shows the aluminium bulge depth measuring device in which the centre point has a small hole to be fitted with a digital depth ruler. Thinning of the plate was measured by ultrasonic thickness testing of the plate at its centre after testing and subtracting this figure from the thickness of the un-deformed plate, as measured prior to blasting.
Chapter 5 – Effects of dynamic impact loading on TWIP steel

Figure 5-1 Aluminum bulge depth gauge.

Figure 5-2(a) shows a schematic diagram of the deformed TWIP steel plate. Six specimens with 10 x10 mm dimension were taken at 70 mm interval from the center of the plate. The numbering of the specimens is starting from the center as specimen 1 to the furthest as specimen 6. Micro- and macro-hardness testing were undertaken using a fully automatic hardness tester, Struers DuraScan-70, with a 0.5 kg and 10 kg load to determine the hardness effect with various specimens. It is notable in Figure 5-2(b) that the horizontal and transverse directions of the specimens were determined by the charge position. The microstructure was analyzed by both scanning electron microscope (SEM) and the electron back scattered diffraction (EBSD) technique which were conducted on, as shown in section 3.3.5, JEOL 6940 and JEOL JSM-7001F field emission gun (FEG) fitted with a Nordlys–II(S)
camera and the EBSD software, AZtecHKL at 15 kV, ~3.3nA and 24 mm working distance. An area of 300x230 µm² was scanned for those six specimens. Post processing of the obtained data was statistically analysed by HKL Channel-5 software.

Figure 5-2 Schematic diagram of (a) one quarter of the bulged plate (upside down) and (b) the transverse and the pressure wave direction determined by the position of the charge. The bulge depth of the deformed plate in this case was 166 mm.
5.3 Results and discussion

5.3.1 Hardness measurement

The micro- and macro-hardness test was performed with HV 0.5 and 10 kg loads, respectively. The values of hardness are presented in Figure 5-3. It is notable that, with two different loads of hardness tests, there is a tendency of declining value with the specimen away from the centre (specimen 1). The macro-hardness of specimen 6 shows almost identical value to the original hardness of the TWIP steel (230 HV) which implies that this area was not affected significantly by the impact. From micro-hardness test, specimen 5 showed an increase of the hardness. This was corresponded to ‘C’ position in Figure 3-3 where an additional impact would be added by the geographical constraint of the die block (between sample 4 and sample 5). This observation was well matched with the Saleh’s work [104] when the specimen numbering system in increasing order was compared with the corresponding annealing temperatures in which higher annealing temperatures resulted in larger grain size.
5.3.2 SEM results

Figure 5-4 shows the SEM images of 6 specimens. It is clear in the figure that with increase of specimen number, the grain size is increased. In another words, less damaged areas clearly have larger grain size. However, the microstructure of the EBT TWIP steel can’t be determined since the interaction between the mechanical twinning and dislocation slips cause the mixing up of the twin and grain boundaries. Therefore, the EBSD technique was further adopted to analyse the microstructure in detail.
Figure 5-4 SEM images of the EBT specimens.

5.3.3 EBSD results

EBSD grain boundary images of all the specimens are shown in Figure 5-5. The black lines indicate high angle grain boundaries (HAGBs) with critical
misorientation $> 15^\circ$, the grey lines represents the low angle grain boundaries (LAGBs) with $2^\circ \leq \theta < 15^\circ$ (the misorientations less than $2^\circ$ were disregarded).

The total high angle grain boundaries (THAGBs) consists of HAGB and twin boundaries (TBs). First order twin boundaries (TBs) are defined as $\Sigma 3=60^\circ \langle 111 \rangle$, shown in red lines, while second order TBs are $\Sigma 9=38.9^\circ \langle 101 \rangle$, represented in blue lines, yielding tolerance limit of $6^\circ$ for $\Sigma 3$ and $2.4^\circ$ for $\Sigma 9$, respectively (following the Palumbo–Aust criterion) [105].

From the observation, specimen 1 has the finest grain size and there is a growing tendency of grain size from Specimen 1 to 6, which is in good agreement with the results of hardness tests. The first order TBs, $\Sigma 3$ (red lines), are evenly distributed in all the samples. A small amount of second order TBs, $\Sigma 9$ (blue lines) is also observed in those six maps. LAGBs were most predominant in Specimen 1 and 2.
Figure 5-5 EBSD grain boundaries map of the EBT specimens.

Each map in Figure 5-5 is composed of scanned data points (pixel); moreover, the data can be statistically analysed to study the microstructure characters and the analysed results are given in Figure 5-6. Figure 5-6(a)
shows the change in the misorientation distribution of the original and EBT specimens and the curve of the theoretical random orientation. Two peaks were observed with representative misorientation axis distribution in the crystal coordinate system. The first order TBs or Σ3 resulted in the intense peak around 60° (related to <1 1 1>), on the other hand, the smaller peak at ~39°, <1 0 1>, represents the Σ9 or second order TBs. It is notable in the Figure 5-6(a) that first order twin (Σ3) boundary area at ~ 60° increases with increasing specimen number. At low misorientation angle, Specimen 1 and 2 revealed high relative frequencies while at high misorientation angle, Specimen 5 and 6 were higher.

Figure 5-6(b) was extracted from the data of Figure 5-6(a). Σ3 boundary area fraction was calculated as the value of correlated frequency of 60° divided by the sum of the frequency. Same method was used to calculate Σ9, THAGBs, HAGBs and LAGBs area fraction. It is also evident in this figure that Σ3 and HAGBs increases with the increase of specimen number which is associated with a decrease in LAGBs and the evolution of Σ9 TBs. Secondary twins are relatively rare for the material in all conditions.
Figure 5-6 (a) Change in misorientation distribution as a function of various specimens with representative misorientation axis distributions in the crystal coordinate system for Σ3 and Σ9 angular ranges, (b) grain boundary area fraction as a function of various specimens, (c) evolution of length fraction of Σ3 and Σ9 twin boundaries with various specimens, and (d) change in grain size with and without taking TBs into account with various specimens.

The boundary with high Σ might be expected to have a higher energy than the one with low Σ. At low-angle boundaries, the distortion is entirely accommodated by dislocations, refer to Figure 5-6(a). For this reason it is concluded that the main strengthening mechanism is work hardening caused...
by grain refinement resulted from the high impact. However, the twin effects cannot be ignored because the dislocations created by the impact will cross over the twin boundaries. This result is clear in Figure 5-6(c). The boundary length fraction increased with the increase of the specimen number. It implies that the existing TBs in more highly deformed regions were consumed by the interaction with dislocations created by the rapid deformation. It is postulated that the first hardening mechanism is dominated by the dislocations created by the impact. The second hardening mechanism is due to the shorter dislocation mean free path cut by deformed twins which turns into more accumulated dislocation. This is in agreement with Kim et al. [25].

Figure 5-6(d) affirms this assumption. The figure shows the change in grain size with and without twin boundaries (TBs). When taking TBs into account, the twin boundaries (both Σ3 and Σ9) were behaving as grain boundaries, therefore, the average grain size is smaller compared with the grain size without taking TBs into account. The grain size without TBs increases with specimen number while the grain size with TBs is steady. The results indicate that the number of twin grain boundaries is evenly distributed over the specimen area, regardless of the original grain size. They also imply that twins may play an important role on this strengthening mechanism. It is
denoted in this figure that the grain size (without TBs) has a trend to increase with the increase of the specimen number, i.e., reduction in deformation even though there was a complex pattern owing to the uneven deformation pattern of the specimens. This is contrary to the results of Li et al. [37] in which the grain size after the deformation is larger than before.

5.3.4 Texture analysis

To study the development of crystallographic texture as a result of explosive bonding, pole figures were derived from the collected results of EBSD data. In the form of stereographic projections, a pole figure is a two dimensional graphical representation of the orientation distribution of crystallographic lattice planes in the material. In this example, (1 1 1) pole figures were displayed representatively in the current investigation. Figure 5-7(a) shows the typical rolling texture components in FCC alloys [107] as reference, consisting of ideal orientation components, including Cube {1 0 0}<0 0 1>, Goss {1 1 0}<0 0 1>, Brass {1 1 0}<1 1 2>, Copper {1 1 2}<1 1 1>, and S {1 2 3}<6 3 4>.

Figure 5-7(b) displays the (1 1 1) pole figure of the original Fe-18Mn-0.6C-1.5Al steel, while Figure 5-7(c-h) exhibit EBT Specimen 1 to 6, respectively. It can be inferred that for the original specimen, the texture
component was mainly cube type \( [0 \ 0 \ 1] \langle 1 \ 0 \ 0 \rangle \) with minor copper type \( [1 \ 1 \ 2] \langle 1 \ 1 \ 1 \rangle \) which possess greater Schmid’s factor of dislocation slip [9].

The pole figure of Specimen 2, 3 and 5 revealed the similar pattern of pole figure to the original (un-impacted) pattern. The pole figures of all other specimens did not show consistent patterns but random ones. This is due to the both the non-directional deformation and the huge energy released during blast experiments. These EBT texture results show significant difference with compression tested samples, as shown in Figure 4.7, which is due to the variance of the deformation methods, one is non-directional, radial explosion and the other is uniaxial compression.
Chapter 5 – Effects of dynamic impact loading on TWIP steel

Figure 5-7 (1 1 1) pole figures of the (a) ideal rolling texture components in FCC materials, and (b) original, (c) Specimen 1, (d) Specimen 2, (e) Specimen 3, (f) Specimen 4, (g) Specimen 5, (h) Specimen 6.
5.4 Conclusions

The EBT was successfully conducted on high manganese TWIP steel and this pre-blast technique posed a potential means of strengthening the material, which mechanism was grain refinement caused by the high impact.
Chapter 6

CPFEM simulation of nano-indentation

The Crystal Plasticity Finite Element Method (CPFEM) has been utilized to simulate the nano-indentation of single crystal high manganese TWinning Induced Plasticity (TWIP) steel in Abaqus environment through the User-Defined Subroutine (UMAT). Two sets of nano-indentation tests were performed on the annealed TWIP steel with Berkovich tip and the crystallographic orientation was examined by means of Electron BackScattered Diffraction (EBSD). By adjusting the input parameters in UMAT, the CPFEM model can be verified by comparing the simulated and experimental load-displacement curves.

6.1 Introduction

The exceptional energy absorption ability has placed the high manganese TWIP steels aiming for the next generation of auto-related materials as well as the military applications [4, 116] where crashworthiness is a vital design factor. This unique ability is resulted from the outstanding combination of properties, ultimate strength and ductility, which are ascribed to the formation of mechanical twins during deformation [30, 51, 117] which twin
boundaries, acting as grain boundaries that blocking the dislocation slips, contribute to enhancement of the work hardening [118]. In other words, the mean free-path of dislocation glide was cut by mechanical twins, which leads to intense strain hardening. Significant works has been dedicated to the development and optimisation of TWIP steels [6, 7, 9, 11, 108, 119, 120] and some physical models have been developed to correlate the strain hardening behaviour along with TWIP effects [9, 11, 24, 121]. Recent attention has been paid to several modelling work, including a “full-field” crystal plasticity finite element method (CPFEM), a “mean-field” multi-site model [122] and ViscoPlastic Self-Consistent (VPSC) plasticity model to simulate the texture and hardening behaviour [5, 6, 123, 124]. However, all aforementioned simulations are the representation of polycrystalline aggregate.

The CPFEM has been implemented to simulate deformation of materials [125-128]. Based on a crystal plasticity constitutive model, it collaborates with the user material subroutine (UMAT) of the commercial finite element software ABAQUS 6.9. Built on the dislocation slip mechanism, the single crystal or polycrystal of body-centred cubic (BCC), face-cantered cubic (FCC) and hexagonal closed packed (HCP) structures will react to an applied stress, which is modelled and simulated by CPFEM. During deformation, grains orientations alter. Simultaneously the threshold stress of each slip system
will increase, which is due to the self-hardening and latent hardening of the deformation patterns. As a result, the CPFEM will record the mechanical response and the orientation of the crystals (texture). By comparing the experimental results with simulated ones, one can get better understanding about texture, stress-strain behaviour, etc., of metal deformation at the grain scale. The CPFEM has been developed insensitively to study the plastic deformation behaviour of materials, including single crystal copper [129-132], single crystal aluminium [131, 133-138], bicrystals aluminium [139], and polycrystal aluminium [140].

Micro- and Nano-indentation are two of the most popular means utilized to investigate the mechanical properties of materials at micro-scale level. Significant Efforts [129, 141-143] have been dedicated to both techniques to study the load-displacement curves, Young’s modulus, size effects etc. The nano-indentation was conducted here to study the hardness and elastic modulus. In this chapter, this is the first time that single crystal TWIP steel was investigated experimentally and numerically. A 3D CPFEM model incorporated with crystal plasticity constitutive equation was used to simulate the process of nano-indentation of and the UMAT parameters were determined by repeatedly comparing with the experimental and simulated results. The surface profile, texture, rotation angle were further investigated.
6.2 Experimental

The description of the TWIP is detailed in chapter 3.1. The as-received material had been hot rolled from slab state and possessed a final thickness of 2.24 mm, with average grain size of approximately 20 μm. The test specimen were cut to a rectangular shape of 4×5×2.24 mm$^3$ and annealed at 1150 °C for 30 minutes. The purpose of annealing is to ensure the enlarged grain size is big enough to accommodate the subsequent nano-indentation experiment to fulfil the assumption of single crystal theory, in other words, a successful nano-indentation experiment should locate within a single TWIP steel crystal; which is to investigate the anisotropy and the micro-scale behaviour under nano-indentation, and to eliminate the effects of grain boundaries and second phase particles. The testing surface was prepared mechanically to the stage of colloidal silica. The nano-indentation experiments were conducted on the IBIS/UMIS system, shown in chapter 3.2.4, with a 200 nm radius Berkovich indenter. Two level of forces, 50mN and 80mN, were conducted. The load-displacement curve for each experiment was derived from penetration on loading and elastic recovery of the indentation on unloading.
After nano-indentation experiments, the initial orientation of each single TWIP steel crystal was measured by a JEOL JSM-7001F, detailed in chapter 3.3.5, with the EBSD software, AZtecHKL at 15 mm working distance. Figure 6-1 shows a representative SEM image of the TWIP steel surface indented by 80 mN load (tilted 70° ready for EBSD scan). The obtain Euler angles \((\phi_1, \Phi, \phi_2)\) were then converted to Miller indices, hkl and uvw, by the following equations,

\[
\begin{align*}
    h &= \sin \Phi \sin \varphi_2 , \\
    k &= \sin \Phi \cos \varphi_2 , \\
    l &= \cos \Phi , \\
    u &= \cos \varphi_1 \cos \varphi_2 - \sin \varphi_1 \sin \varphi_2 \cos \Phi , \\
    v &= -\cos \varphi_1 \sin \varphi_2 - \sin \varphi_1 \cos \varphi_2 \cos \Phi , \\
    w &= \sin \Phi \sin \varphi_1 .
\end{align*}
\]

The Miller indices were used as input parameters in the user material card.

Figure 6-1 SEM image of the Fe-18Mn-0.6C-1.5Al TWIP steel surface after 80mN load of nano-indentation (tilted 70° ready for EBSD scan).
6.3 Finite element implementation

The crystal plasticity model coded in the UMAT is built on an assumption that, in all activated slip systems, plastic deformation is the sum of the crystalline slip, which has been utilized widely to explain the plastic deformation of single crystals [134, 136, 144]. With a given starting crystalline orientation and material parameters of the model, the CPFEM is able to calculate the stress-strain response and texture evolution of crystals. In current study, the line described by Asaro [126] was implemented to a crystal plasticity constitutive model incorporated with the implicit finite element program ABAQUS/Standard via the UMAT. The important functionalities of UMAT are to offer the Jacobian matrix of material, \( \partial \Delta \sigma / \partial \Delta \varepsilon \) for the constitutive model and to update the stress and the solution dependent state variables including crystal orientation. In this study, we adopted the UMAT framework developed by Huang [145] and utilized Asaro’s formulation as the hardening model. Since the latent hardness has been taken into account, the tangent stiffness matrix (Jacobian matrix) is not symmetric. In addition, “unsymmm” must be checked in the input file at the user material card.

The deformation of nano-indentation was simulated by the commercial software Abaqus6.9. A 3-dimensional, \( 30 \times 30 \times 20 \mu m^3 \) in size, model was
constructed to illustrate the procedure of the deformation, as shown in Figure 6-2. The X, Y, and Z coordinates indicate the rolling direction (RD), the transverse direction (TD), and the normal direction (ND), respectively. The initial orientation was obtained from the EBSD experimental data. The height (20 µm) is about 20 times larger than the maximum displacement of the nano-indentation due to the rule that indentation depth should be 10 times smaller than the thickness of the specimen to avoid the influence from the substrate. Another software, Solidworkds, was used to create the 200 nanometres radius Berkovich indenter which was then imported into Abaqus environment. In order to assure the fineness of the mesh, the specimen was composed of 15336 eight-node brick elements and 16701 nodes with reduced integration (element id: C3D8R). Finer mesh was used in the contacting area around the Berkovich tip. The quantity of elements and nodes is about 6 times more than those used in recent studies [130, 146] since the deformations of those studies are smaller (300 nm in depth) than current study (1000 nm).
The time step increment was set for the convergence of modelling and a fixed time step increment of 0.01 s was adopted, which resulted in a total time step increments of 24037, including the steps of contact, loading and unloading. All the nodes on the bottom surface of the specimen were constrained according to the nano-indentation experimental testing condition (the bottom of the sample is glued on the stage). As the coefficient of friction doesn’t affect the load-displacement curve significantly [130], it was not considered in this study.
6.4 Results and discussion

The UMAT input parameters are listed in two groups: elastic and plastic parameters. For the elastic parameters, the elastic constants $C_{11}, C_{12},$ and $C_{44}$ are adopted instead of the elastic modulus $E$ and Poisson’s ratio $\nu$ due to the single crystal assumption. The elastic constants $C_{11}, C_{12},$ and $C_{44}$ are obtained from a recent work of Pierce [147] as 169, 82 and 96 GPa respectively. There are three types of plastic parameters: viscoplasticity parameters, $n$ (strain rate sensitivity exponent) and $\dot{\alpha}$ (reference strain rate); self hardening parameters, $h_0$ (initial hardening modulus), $\tau_s$ (saturation stress), and $\tau_0$ (initial critical resolved shear stress); latent hardening parameter, $q$. The plastic parameters were to be identified by fitting method. that is to say, by matching the simulated load-displacement curve with the experimental one, the correct input parameters can be acquired.

The effect of the $n, \dot{\alpha}, h_0, \tau_s,$ and $\tau_0$ are shown in Figure 6-2, 6-3, 6-4, 6-5, and 6-6 respectively.
Figure 6-3 Effect of \( n \) on the numerical load-displacement curves.

Figure 6-4 Effect of \( \dot{\alpha} \) on the numerical load-displacement curves.

\( n \) doesn’t have significant effect on the simulated curves while \( \dot{\alpha} \) does which might be due to the increasing magnitude of individuals. However, Zambaldi an Raabe reported that the strain rate sensitivity exponent \( n \) and
the reference strain rate $\dot{\varepsilon}$ are assumed to be constants since the principal meaning of they are of numerical nature instead of having relationship with mechanical properties [148]. Along with latent hardening parameter $q$, those parameters are obtained from literature as common values for CPFEM [129, 149-151].

![Graph showing the effect of $h_0$ on the numerical load-displacement curves.](image)

Figure 6-5 Effect of $h_0$ on the numerical load-displacement curves.

As observed in Figure 6-5, the maximum load increases as $h_0$ decreases. On the other hand, increasing $\tau_s$ and $\tau_0$ values resulted in evaluated values of maximum load as shown in Figure 6-6 and 6-7. The fitting method was to determine the appropriate $h_0$, $\tau_s$ and $\tau_0$ for matching the experimental and simulated load-displacement curves for both levels of loads (50 and 80 mN).
The fitting process was lengthy and time consuming. The determined UMAT input parameters are given in Table 6-1.

![Figure 6-6 Effect of \( \tau_s \) on the numerical load-displacement curves.](image)

![Figure 6-7 Effect of \( \tau_0 \) on the numerical load-displacement curves.](image)
Table 6-1 Parameters used in the CPFEM simulation.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Physical meaning</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$C_{11}$</td>
<td>Elastic constant (MPa)</td>
<td>169000</td>
</tr>
<tr>
<td>$C_{12}$</td>
<td>Elastic constant (MPa)</td>
<td>82000</td>
</tr>
<tr>
<td>$C_{44}$</td>
<td>Elastic constant (MPa)</td>
<td>96000</td>
</tr>
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<td>$h_0$</td>
<td>Initial hardening modulus (MPa)</td>
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<tr>
<td>$\tau_s$</td>
<td>Saturation stress (MPa)</td>
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<tr>
<td>$\tau_0$</td>
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<td>$n$</td>
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<tr>
<td>$\dot{\varepsilon}$</td>
<td>Reference strain rate (/s)</td>
<td>0.001</td>
</tr>
<tr>
<td>$q$</td>
<td>Latent hardening parameter</td>
<td>1</td>
</tr>
</tbody>
</table>

6.4.1 Young’s modulus

Figure 6-8 and 6-9 gives the load-displacement curve [152] of the experimental and CPFEM simulated data of (a) 50 mN, and (b) 80 mN loads, respectively. The indentation experiment was conducted on the plane which initial orientation was obtained from EBSD experiment. The experimental and simulated results from both 50 and 80 mN are matched well.
Figure 6-8 Experimental and Simulated load-displacement curves of 50 mN load.

Figure 6-9 Experimental and Simulated load-displacement curves of 80 mN load.
By adopting the following equations from Oliver and Pharr [153, 154], the Young’s modulus could be derived from the curves shown in Figure 6-3.

\[
h_c = h_{\text{max}} - k \frac{p_{\text{max}}}{S}, \quad k = 0.75, S = \left(\frac{dp}{dh}\right)_{h=h_{\text{max}}} \quad (6 - 1)
\]

\[
A_c = 3\sqrt{3}h_c^2\tan^2 65.3 = 24.56h_c^2 \quad (6 - 2)
\]

\[
E_r = \frac{dp}{dh} \frac{1}{2h_c} \frac{1}{\beta} \sqrt{\frac{\pi}{24.5}}, \quad \beta = 1.034 \quad (6 - 3)
\]

\[
\frac{1}{E_r} = \frac{1-v^2}{E} + \frac{1-v_i^2}{E_i} \quad (6 - 4)
\]

where \(h_c\) is the contact depth and the \(k\) factor is related to the indenter. For the spherical indenter, including Berkovich, \(k = 0.75\). For the conical indenter \(k = 0.72\). \(S\) represents the slope of the unloading curve and \(A_c\) denotes the contact area. \(\beta\) depends upon the indenter and the value is 1.00, 1.034, and 1.012 for the conical, Berkovich, and Vickers respectively. \(E_r\), \(E_i\) and \(E\) represents the Young’s modulus of the residual, the diamond indenter, and the measured specimen, respectively. \(v\) is the Poisson’s ratio which value is 0.27-0.30 for steel, but 0.28 for this particular TWIP steel [67]. \(E_i = 1141\text{GPa}\) and \(v_i = 0.07\) [153]. From Figure 6-8, the Young’s modulus, for 50mN load, was calculated from the experimental and simulation curves as 181 GPa and
211.7 GPa, respectively. Similarly, for 80mN load, the values of experimental and simulation are 192.2 GPa and 219.5 GPa obtained from the curves of Figure 6-9. The difference between the measured Young’s modulus at different load is due to surface influence. The results of Young’s modulus are matched well with literature [64, 67].

6.4.2 Surface profile

After unloading of 50mN, the simulated surface profile in the Z surface along the diagonal and vertical cutting lines, the red lines on Figure 6-10(a), are shown in Figure 6-11(a-b). Similarly for 80 mN, the simulated surface profile in the Z surface along the diagonal and vertical cutting lines, the red lines on Figure 6-10(b), are shown in Figure 6-11(c-d). The surface profiles along the diagonal line shows the piling-ups for both levels of loads. The same phenomenon was found in vertical cutting line as well. The piling-up increases as the load increases. The higher nano-indentation force (80 mN) resulted in enhanced magnitude of the pile-ups. From both diagonal and vertical cutting line, higher piling-up can be found at the face side of the Berkovich indenter rather than the tip which is in agreement with Liu et al. [138].
Figure 6-10 Distribution of out-of-plane displacement after (a) 50 mN and (b) 80 mN unloading.
Figure 6-11 Comparisons of the surface profile after unloading for 50 mN loads along (a) diagonal and (b) vertical directions and for 80 mN loads along (c) diagonal and (d) vertical directions.

6.4.3 Pole figure

The pole figure was acquired from underneath the left side indenter of simulated model. Figure 6-12 and 6-13 represents the evolution of the pole figure of two levels of nano-indentation loading forces, 50 mN and 80 mN, from (a) initial (before deformation) to (b) halfway of the loading, (c) final step of loading, and (d) final step of unloading. For both Figure 6-12 and
6-13, there is not much difference found between (c) and (d) pole figures while less texture effect was found in (b).

Figure 6-12 Pole figures representation of (a) initial, (b) halfway of loading, (c) final step of loading, and (d) final step of unloading for 50 mN load.

Figure 6-13 Pole figures representation of (a) initial, (b) halfway of loading, (c) final step of loading, and (d) final step of unloading for 80 mN load.

6.4.4 Lattice rotation angles

Figure 6-14 shows the lattice rotation angles around the RD after unloading of (a) 50 mN and (b) 80 mN forces. The cross section is acquired from the red vertical cutting line in Figure 6-10. The lattice rotation angle is composed of three components, including the angle around the X, Y and Z axis.
individually. The calculation of the rotation is followed Wert et al. [155].

From Figure 6-14, the biggest rotation angle was found on the sharp angle side of the indenter rather than the face side. And the positive values were found on angle side while the negative ones found on face side.

![Figure 6-14](image)

Figure 6-14 Comparisons of the lattice rotation angle after unloading of (a) 50 mN and (b) 80 mN forces.

6.5 Conclusions

A crystal plasticity FEM model has been developed to simulate the process of nano-indentation and the crystal orientation was obtained from EBSD technique. The UMAT input parameters for TWIP steel have been determined via fitting method by matching the experimental and simulated load-displacement curves with different loads where \( h_0 \) (initial hardening
modulus) has the positive correlation on the curves while \( \tau_s \) (saturation stress) and \( \tau_0 \) (initial critical resolved shear stress) has negative one. All the calculated Young’s modulus values are in good agreement with the literature. The surface profile was generated as well as the pole figure and lattice rotation angles. The piling-up was found in all cases. The positive values of the rotation angle were found on the long side of the indenter while the negative ones were found on the face side.
Chapter 7

Conclusions

The mechanical properties obtained from compression tests revealed the superior properties of this Fe-18Mn-1.5Al-0.6C TWIP steel and the microstructural and crystallographic characterization indicated that deformation twinning was the main reason for the excellent strain hardening behaviour. Compression supresses the activity of twinning as compared to tensile loading. Mechanical twinning plays a major role during deformation while slip also makes a contribution.

A pre-blast technique was demonstrated to be a feasible method of strengthening the TWIP steel. The mechanism behind is grain refinement resulted from high impact loading. The generation of twin boundaries (TBs) acting as barrier blocking the movement of slip which posing an alternative means of strengthening.

By combining the nano-indentation, EBSD experiments and CPFEM modelling with Abaqus, three unknown UMAT input parameters were determined by fitting method, in other words, by repeatedly adjusting the
parameters and verifying the experimental and simulated load-displacement curves, the appropriate input parameters were acquired. The obtained UMAT parameters can be used for further modelling work to simulate other deformation of this TWIP steel, for example, tensile test reported in literature, compression and EBT tests performed in Chapter 4 and 5.
References

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