Micromechanics of Twinning in a TWIP Steel

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Abstract

The deformation behaviour of a TWinning Induced Plasticity (TWIP) steel was studied at quasi-static strain rates using synchrotron X-ray diffraction. A $\{111\}$ RD and $\{200\}$ RD texture developed from the earliest stages of deformation, which could be reproduced using an elasto-plastic self consistent (EPSC) model. Evidence is found from multiple sources to suggest that twinning was occurring before macroscopic yielding. This included small deviations in the lattice strains, $\{111\}$ intensity changes and peak width broadening all occurring below the macroscopic yield point. The accumulation of permanent deformation on sub-yield mechanical cycling of the material was found, which further supports the diffraction data. TEM revealed that fine deformation twins similar to those observed in heavily deformed samples formed during sub-yield cycling. It is concluded that twinning had occurred before macroscopic plastic deformation began, unlike the behaviour traditionally expected from hexagonal metals such as Mg.

Key words: Twinning, Synchrotron Radiation, Austenitic Steel, Yield Phenomena, Micromechanical Modeling

1. Introduction

High manganese content steels deform through the evolution of mechanical twins, and have therefore become known as TWinning Induced Plasticity (TWIP) steels. These austenitic steels are generally based on the Fe-Mn-Si-Al-C system, where the composition is adjusted to tailor the stacking fault energy (SFE) to be within a desired range [1]. Manganese and aluminium additions raise the SFE, while the addition of silicon, in sufficient amounts acts to reduce the SFE [5, 6]. TWIP steels exhibit extremely high strain to failure, up to 95 %, at tensile strengths in excess of 800 MPa [1, 2, 3]. This extraordinary combination of properties has led to significant interest in using these steels in

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high strain rate energy adsorption applications, such as automotive crash safety systems and military vehicle armour [7].

The SFE is affected by temperature and composition, consequently the value is of great practical importance in face centred cubic (*fcc*) materials, particularly austenitic steels. The SFE influences the processes of dislocation cross-slip and climb [4], which have an important role in the work hardening behaviour of a material. Low SFE results in stacking faults becoming wider, thus making dislocation cross-slip more difficult. Therefore it is essential to tailor the alloy composition for a desired SFE range.

Austenitic steels deform *via* a phase transformation, mechanical twinning or dislocation glide depending on the SFE [8]. At high energies ($\geq 45 \text{ mJ m}^{-2}$) deformation progresses solely by dislocation glide. Lower SFE ($\leq 18 \text{ mJ m}^{-2}$) promotes a martensitic transformation to ϵ -martensite, $\gamma_{fcc} \rightarrow \epsilon_{hcp}$, which becomes a two step transformation to form α' -martensite at even lower energies, $\gamma_{fcc} \rightarrow \epsilon_{hcp} \rightarrow \alpha'_{bcc}$ [9]. A SFE ranging between 18 - 45 mJ m^{-2} promotes the formation of mechanical twins during deformation, which enables excellent ductility to be achieved.

Deformation twinning is a process which proceeds through a dislocation mechanism. Mahajan [10] proposed two salient features of deformation twin formation in *fcc* crystals; firstly a three layer twin may nucleate when two co-planar $\frac{1}{2}\langle 110 \rangle$ dislocations interact, and secondly a macroscopic twin may evolve when three layer twins at different levels grow into each other. Two co-planar perfect $\frac{1}{2}\langle 110 \rangle$ dislocations can react either through co-planar slip or cross glide, thus the reaction; $\frac{1}{2}[\bar{1}01] + \frac{1}{2}[\bar{1}10] \rightarrow 3 \times \frac{1}{6}[\bar{2}11]$, is possible. It is believed that this reaction governs the nucleation of twins in *fcc* crystals. Furthermore in low stacking fault energy materials that twin more readily, the perfect dislocations will dissociate into Shockley partials; $\frac{1}{2}[\bar{1}10] \rightarrow \frac{1}{6}[\bar{2}11] + \frac{1}{6}[\bar{1}2\bar{1}]$. This is believed to be an intermediate step to the previous reaction as it is energetically favourable. Consequently a macroscopic twin forms when the nucleated twins grow into each other, *i.e.* absorb more partials. This may explain why twinning is less favourable in materials with high stacking fault energies, because the partials are more tightly bound *i.e.* the partial dislocation pair have a narrow core width and lower mobility. The self-thickening of twins has recently been observed *in-situ* in coarse grained Cu-Ge alloys and nanocrystalline Ni [11, 12]. A perfect dislocation was found to dissociate into leading and trailing partials. The leading partial subsequently splits further into a perfect dislocation and twinning partial upon encountering an obstacle. Subsequently the perfect dislocation can cross-slip and dissociate once again. This cycle continues, thereby thickening the twin on successive primary slip and cross-slip planes, without the need for the nucleation of additional Shockley partials.

Twins are observed by the formation of discrete sheared grain subregions characterised by a mirror plane at the twin interface. Consequently, the high strength and ductility of TWIP steels is due to the formation of preferentially localised twins, which lead to extensive local strain hardening. Further twinning results in an increase in the volume fraction of twins leading to a marked decrease in the mean free path for dislocation movement. This results in the

characteristic high levels of hardening in the material. These properties mean that TWIP steels are able to meet the requirements of high formability and energy absorption.

Although research into TWIP steels is increasing, the evolution of microstructure and grain statistics, such as elastic lattice strain and texture, especially during deformation have not been investigated in detail. Therefore limited information is available within this area. Microstructure and texture evolution during deformation has been investigated by a few authors [13, 14, 15], however this has been achieved using *ex-situ* interrupted testing and multiple samples. Consequently, the characterisation of the twins and texture has taken place after testing and only limited bulk information could be determined. Yan *et al.* [16] have used *in-situ* synchrotron diffraction to characterise the interaction between slip and twinning during uniaxial tension. It was determined that the deformation texture is predominantly developed by dislocation gliding and that twinning impedes the reinforcement of texture.

Eshelby's [17, 18] self consistent approach has successfully been used to simulate the uniaxial deformation texture and lattice strain behaviour of several materials [19, 20, 21, 22]. However, the application of self-consistent modelling to TWIP steels has been rather limited. Prakash *et al.* [23] have utilised the visco-plastic self-consistent (VPSC) model framework to evaluate twin volume fraction and compare experimental and simulated deformation texture using two different twinning models. A predominant twin reorientation [24] model and the Kalidindi [25, 26] approach were both evaluated, and the latter was found to predict more plausible twin activity and provide better agreement with experimentally observed textures. Experimental texture and grain statistics have also been modelled by Yan *et al.* [16] using both a VPSC and elasto-plastic self-consistent (EPSC) models. The VPSC model was utilised to simulate the experimental textures, but the model textures indicated an over prediction for the contribution of twinning in the simulation. Similarly the EPSC model was used to evaluate the experimental lattice strain evolution. However, the EPSC simulation did not include a twinning scheme, consequently the contribution of slip during deformation was only modelled for the lattice strain analysis.

In the present work, the texture evolution of a TWIP steel during tensile deformation has been investigated using *in-situ* synchrotron X-ray diffraction. Lattice strain, peak width and intensity changes have also been examined, and the elasto-plastic self consistent model [20] has been utilised to rationalise the results. *Ex-situ* microscopy and cyclic tensile loading experiments were also conducted, to augment the *in-situ* observations.

2. Experimental Procedures

The TWIP steel tested (Fe-0.7C-2Al-15Mn-2Si wt %) was obtained in 3 mm rolled sheet form from Tata Steel Strip Mainland Europe. The stacking fault energy of the material was determined using thermodynamic calculations to be $31 \pm 10 \text{ mJ m}^{-2}$ with adjustments made to account for the silicon and aluminium contents [27, 28, 5].

2.1. In-situ synchrotron diffraction

In-situ testing was carried out on beamline ID15B at the European Synchrotron Radiation Facility (ESRF), Grenoble, France. Tensile samples, with gauge dimensions of $19 \times 1.5 \times 1.5$ mm, were tested on an Instron 5 kN servohydraulic machine, with the tensile axis aligned to the rolling direction of the material. Samples were held in bespoke negative profile fixtures and loaded to an engineering strain of 22 % in position control and at an initial strain rate of 10^{-3} s^{-1} . Full Debye-Scherrer diffraction rings were collected using a $300 \times 400 \mu\text{m}$ monochromated X-ray beam of energy 67 keV ($\lambda = 0.1428 \text{ \AA}$) on a Pixium 2D area detector located 1046 mm from the sample. A sampling time of 0.4 s was used. The experimental setup was identical to that used in Ref [29] and testing was conducted consecutively, a schematic representation of the setup is shown in Figure 1.

The texture during deformation was reconstructed by segmenting the diffraction rings into intensity- 2θ profiles using 10° interval bins around the whole ring, using the program Fit2D [30]. Instrumental parameters were obtained by using a CeO_2 powder standard. The intensity- 2θ profiles were then fitted through Rietveld refinement and the texture was plotted using an Extended-WIMV (E-WIMV) algorithm *via* the Materials Analysis Using Diffraction (MAUD) [31] program. Pole figures were visualised using the programs Pole8 and Pod2k.

Figure 1: Schematic representation of the experimental setup for *in-situ* loading experiments. Figure adapted from Ref [29].

Lattice strain is represented by the lattice spacings therefore the elastic strain in a certain direction for a peak $\{hkl\}$ satisfying the diffraction condition can be determined by finding the lattice parameter d_i^{hkl} using Bragg's law, $\lambda = 2d \sin \theta$, relative to an unstrained reference lattice parameter, d_0^{hkl} . Movement of the diffraction peaks can then be related to the elastic strain within grains in the diffraction orientation by

$$\epsilon_{hkl} = \frac{d_i^{hkl} - d_0^{hkl}}{d_0^{hkl}} \quad (1)$$

127 Therefore the shift of an $\{hkl\}$ peak along the loading direction is a measure of
128 strain in that $\{hkl\}$ orientation.

129 To determine the lattice strains, individual diffraction peaks were fitted util-
130 ising a Gaussian function with the Wavemetrics program Igor Pro. d -spacing
131 and intensity values for each peak were obtained from the 0 and 180° tensile
132 loading bins.

133 2.2. *Ex-situ tensile testing*

134 *Ex-situ* tensile tests were carried out on a Zwick Roell 100kN load frame
135 and samples with gauge dimensions of $40 \times 8 \times 3$ mm were tested, with the
136 tensile axis aligned to the rolling direction, using an extensometer. Samples
137 were cyclicly loaded between 10 MPa and a selected target stress 20 times un-
138 der position control at a strain rate of 10^{-3} s^{-1} . The initial target stress was
139 200 MPa, this was incrementally raised by 100 MPa after the completion of 20
140 cycles until the sample failed.

141 2.3. *Ex-situ characterisation*

142 Samples for optical microscopy were etched using 4% Nital to reveal the
143 grain boundaries. A standard metallographic preparation schedule was followed.

144 EBSD was performed on a JEOL JSM6400 SEM equipped with an Oxford
145 Instruments HKL Nordlys EBSD detector. Step sizes between 0.1 and 0.5 μm
146 were employed and in excess of 3000 grains were indexed for texture measure-
147 ment. XRD texture measurements were made using a Philips XPert MRD
148 machine in texture configuration *via* the back reflection technique using copper
149 K_α radiation. The data was then reconstructed onto equal-area projection pole
150 figures using the preferred orientation package - Los Alamos (popLA) [32] tex-
151 ture analysis software package. The Philips XPert MRD machine can measure
152 pole intensities up to an angle of 80° from the sample plane normal, therefore a
153 correction for defocussing was performed to complete the outer ring of the pole
154 figures.

155 TEM analysis was conducted on a JOEL 2000FX microscope; samples were
156 electropolished using 5 vol.% perchloric acid and 95 vol.% acetic acid at 30 V
157 DC and at room temperature.

158 3. EPSC Simulation

159 3.1. *Self-consistent modeling*

160 The elasto-plastic self consistent (EPSC) model follows the single crystal
161 plasticity model described by Hutchinson [18], which is based on the work con-
162 ducted by Hill [33, 34]. The EPSC approach treats each individual grain as
163 an ellipsoidal elasto-plastic inclusion embedded within a homogeneous effective
164 medium (HEM), representing the polycrystalline aggregate. The calculation
165 procedure involved in the EPSC model is explained in detail elsewhere [20],
166 therefore only a brief summary of the fundamental equations used in the model
167 will now follow. Eshelby demonstrated that the stress and strain primarily

within an ellipsoidal inclusion is constant and proportional to the applied stress and strain. Although the problem is elastic, it can be extended to the plastic regime by expressing the solution in an incremental form and introducing the concept of the instantaneous modulus, which relates the stress rate, $\dot{\sigma}$ to the total strain rate, $\dot{\epsilon}$. This forms the constitutive equation for the medium; $\dot{\sigma} = L : \dot{\epsilon}$, where L is the overall instantaneous elasto-plastic stiffness. Similarly the constitutive equation for a particular grain is $\dot{\sigma}^c = L^c : \dot{\epsilon}^c$, where the superscript c denotes an individual grain. The grain modulus L^c is dependent on the orientation of the grain, single crystal elastic constants and plastic state of the grain.

Eshelby showed that the total strain rate in a grain can be related to that in the bulk medium *via* $\dot{\epsilon}^c = A^c : \dot{\epsilon}$, where $A^c = (L^c + L^*)^{-1} : (L + L^*)$, L^* is the effective stiffness, determined by $L^* = L : (S^{-1} - I)$, where S is the elasto-plastic Eshelby tensor and I is the fourth order identity tensor.

The total strain rate relationship provides the strain rate in an individual grain for a given medium stiffness and imposed strain rate. But, L is dependent on the response of individual grains, therefore it has to be determined iteratively with the condition that the strain of the medium is equal to the weighted average over all the grains $\dot{\epsilon} = \langle \dot{\epsilon}^c \rangle$, *thus* the self-consistent equation for the bulk stiffness can be obtained; $L = \langle L^c A^c \rangle \langle A^c \rangle^{-1}$, while the final equation is solved iteratively. Ellipsoids of different shapes and orientations can be included.

3.2. Simulation of TWIP steel

The deformation of the TWIP steel was simulated to match experimental conditions, namely; a strain rate of 10^{-3} s^{-1} to an engineering strain of 22%. The texture was modelled from a starting basis of 1000 weighted grain orientations obtained *via* a sample orientation distribution function using popLA, from the ESRF X-ray synchrotron starting texture measurement of the as-received material.

Twinning and slip were both modelled in the simulation. The hardening behaviour of each slip system was modelled using an extended Vocé law:

$$\tau^s = \tau_0^s + (\tau_1^s + \theta_1^s \Gamma) \left(1 - \exp \frac{-\theta_0^s \Gamma}{\tau_1^s} \right) \quad (2)$$

where τ_0^s is the threshold stress for activation of a deformation mode, τ_1^s is the Vocé stress where the hardening extrapolates to zero, θ_0^s is the initial hardening rate and θ_1^s is the final asymptotic hardening rate for the slip system, s , while Γ is the accumulated shear strain in the grain. In the present simulation, grain rotation due to slip is included, such that the texture can evolve.

Twinning was modelled using the extended EPSC code by Clausen *et al.* [21]. Here twinning is essentially treated as a slip system in the model, whereby a critical resolved shear stress is assigned. However, opposite twin systems are not considered since twinning is unidirectional. A new ‘child’ grain is created in the parent grain when twinning becomes active, the orientation of which is dependent on which twin system is being activated. The twins are allowed

209 to grow and reorientate the parent grain to the new twin orientation, however
 210 this needs to be accounted for in the model, whilst keeping the overall volume
 211 fraction of the parent grain constant. The increment in twin volume fraction
 212 (δw^{twin}) is given by

$$\delta w^{twin} = \frac{\delta \gamma^{twin}}{S^{twin}} \quad (3)$$

213 where $\delta \gamma^{twin}$ is the shear increment contributed by the twin system of the parent
 214 grain and S^{twin} is the characteristic twinning shear. Subsequently the change
 215 in the parent grain volume can be obtained from

$$\delta w^{parent} = - \sum_{twin} \delta w^{twin} \quad (4)$$

216 Once a twin is created the reorientated region is treated as a new independent
 217 grain, furthermore the model allows for a whole grain to be consumed by the
 218 twin.

219 The stress relaxation procedure was activated within the twinning scheme to
 220 account for the contribution to diffraction by the twinned volume fractions and
 221 also provide some degree of stress relaxation in the grains due to twin activity.
 222 The procedure also requires strain compatibility at the twin interface to be
 223 satisfied.

224 Single crystal elastic constants for austenite (γ -Fe) were obtained from the
 225 literature [35] and are listed in Table 1. TWIP steels deform *via* $\{111\}\langle 110 \rangle$
 226 slip and $\{111\}\langle 112 \rangle$ twinning. The critical resolved shear stress (CRSS) for each
 227 deformation mode was selected based on an approximation from the bulk stress-
 228 strain curve, these were then adjusted along with the hardening parameters until
 229 an acceptable fit for bulk stress-strain, texture and lattice strain behaviour was
 230 obtained between the model and experimental results, Table 2.

Table 1: Single crystal elastic constants in GPa for γ -Fe (austenite) [35].

	C_{11}	C_{12}	C_{44}
Austenite (γ -Fe)	276	173.5	136.3

Table 2: Fitted critical resolved shear stress and Voce hardening parameters for each deformation system in GPa.

Mode	Label	CRSS (τ_0)	τ_1	θ_0	θ_1
$\{111\}\langle 110 \rangle$	Slip	0.26	0.56	0.36	0.27
$\{111\}\langle 112 \rangle$	Twinning	0.17	0.45	0.22	0.07

Figure 2: (a) Light micrograph of the as-received material, (b) EBSD map of the initial material [85 % indexing] and (c) weak random texture determined using EBSD, XRD and sXRD.

4. Results and Discussion

4.1. Macroscopic characterisation

The initial microstructure of the material was single phase composed of equiaxed austenite grains, some of which contained annealing twins. The average grain size was $10 \pm 6 \mu\text{m}$, which was determined using EBSD, Figure 2(a,b). The initial texture of the TWIP steel plate was weak and essentially random, Figure 2(b,c), this was confirmed using three experimental techniques, namely electron backscatter diffraction (EBSD), lab X-ray diffraction (XRD) and synchrotron X-ray diffraction (sXRD). The conclusion of an initial random texture from three separate experimental techniques provides a sound basis for the reconstruction of the synchrotron X-ray diffraction data, since it implies that Rietveld refinement of the data does not induce any false texture.

The bulk stress-strain curve for the material tested to 22 % engineering strain is shown in Figure 3. A yield of approximately 460 MPa was observed and the yield transition occurred gradually. The elastic modulus of the material was 170 GPa. An almost linear true hardening rate of $\sim 2.3 \text{ GPa}$ is exhibited, indicating a high level of strain hardening. A high linear hardening rate is often observed in austenitic steels that deform through twinning, as this is usually associated with the interaction between dislocation gliding and twinning [36]. The formed twins act as strong barriers to dislocation motion and further straining results in a greater volume fraction of twins being created. This effectively results in a continuous grain refinement process *i.e.* a dynamic Hall-Petch effect where the mean free path for dislocation gliding is continuously reduced [37, 13].

The tensile behaviour predicted by the model shows excellent agreement with the experimental results and the gradual yielding and linear hardening rate is also well replicated. The predicted activity of the deformation modes suggested a sharp increase in twinning activity at the onset of plastic deformation, which gradually decreases with further deformation. The sharp increase in twin activity may be interpreted as the nucleation of twins in the material when plastic deformation initiates. The slow decrease in activity with further

Figure 3: Macroscopic stress-strain and hardening (inset) curves showing the experimental (solid line) tensile behaviour and the model prediction (dashed line).

261 deformation may indicate a cessation in twin nucleation and the initiation of
 262 twin thickening and subsequent twin growth within the model framework.

263 4.2. Lattice strain and peak width evolution

264 The evolution of lattice strain during loading is shown in Figure 4, along
 265 with the predicted model results. Figure 4 also shows the lattice strain be-
 266 haviour of an austenitic 304 stainless steel, which was tested using an identical
 267 experimental setup during consecutive tests (data from Ref [29]). The $\{111\}$
 268 orientation is the stiffest orientation, which is followed by the $\{220\}$, $\{311\}$ and
 269 $\{200\}$. Furthermore the $\{200\}$ and $\{311\}$ orientations are observed to remain
 270 elastic the longest. This behaviour can be seen more clearly in the simulated
 271 results, Figure 4 (solid black lines). The diffraction elastic constants remain
 272 consistent with that observed in other austenitic steels [38], whereby the order
 273 of the stiffest orientations are as expected. The model successfully predicts the
 274 general behaviour of the orientations, however the simulated results do deviate
 275 from the experimental data. The disparity between the experimental and
 276 simulated lattice strain can firstly be explained due to the changes observed
 277 in the experimental lattice strain below the yield point which are difficult to
 278 replicate within the model. Secondly, the model prediction is a compromise
 279 fit between macroscopic stress-strain, texture and lattice strain and finally the
 280 model predicts twin activation to occur later than the experimental evidence
 281 would indicate. Consequently, simply activating twinning earlier in the model
 282 would result in orientations yielding prematurely and texture strengthening,
 283 however this would not result in the observed sub yield deviations in the lattice
 284 strains.

285 The $\{200\}$ orientation begins to gradually yield below the macroscopic yield
 286 point, which is indicated by the inflections observed at ~ 100 and 200 MPa. This

Figure 4: Experimental and simulated lattice strain evolution during tensile loading of the TWIP steel compared to the lattice strain evolution of an austenitic 304 stainless steel. 304 data from Ref [29], sample texture from prior forming operations prevented diffraction from the $\{220\}$ orientation.

would indicate that micro-plasticity occurs in this orientation before macroscopic yielding. Although micro-plasticity can occur before macroscopic yielding, the experimental results indicate this occurs at a stress significantly lower than the yield point. The $\{111\}$ and $\{220\}$ orientations exhibit a similar behaviour, which suggests the initiation of non-linear behaviour before the macroscopic yield point for all these orientations and micro-plasticity. The onset of plasticity in three orientations at stresses below the yield point cannot be replicated using the self-consistent model and they are not observed in the 304 stainless steel. However the model does indicate plasticity occurs at a slightly lower stress than the yield point. This implies that the material is not truly elastic before the yield point. Small inflections are seen in the lattice strain profiles once the macroscopic yield point is exceeded, this can be replicated using the model and be clearly seen in Figure 4. After macroscopic yielding the grains become plastic and consequently they are no longer able to accrue elastic load at the same rate as when the material was macroscopically elastic. This usually corresponds to an inflection in the lattice strain profile around the yield stress of the material. The inflections are the result of load partitioning from orientations that have plastically deformed to those that have not. The experimental evidence shows that the $\{111\}$ and $\{220\}$ orientations are the first to exhibit such a reduction in the rate of accumulating lattice strain as deformation progresses, while the $\{311\}$ and $\{200\}$ orientations are most capable of accumulating further strain. Therefore as further deformation progresses, grains in the $\{311\}$ and $\{200\}$ orientations accumulate the majority of the corresponding lattice strain. A non-linear increase of lattice strain is clearly apparent for the $\{200\}$ orientation, this orientation is also able to accrue greater lattice strain compared to other orientations, thus further indicating load partitioning between the orientations.

An obvious feature in the experimental lattice strain, Figure 4, are the deviations from linearity, particularly below the macroscopic yield point. This can be clearly seen for the $\{111\}$, $\{200\}$ and $\{220\}$ orientations, where a $-170 \mu\epsilon$ deviation is observed for the $\{111\}$ and a $-740 \mu\epsilon$ is observed for the $\{220\}$ ori-

318 entations at ~ 120 MPa. This value is very similar to the twin initiation stress
 319 predicted for the experimental material by the authors in a separate study [39].
 320 Similar behaviour has been seen in Ref [16], particularly for the $\{220\}$ orienta-
 321 tion. However, the authors attributed this behaviour to be due to error bars
 322 caused by poor diffraction sampling of the grains in the longitudinal direction.
 323 In the present study, it is believed that these deviations are due to intrinsic
 324 changes within the material and not because of poor diffraction sampling. The
 325 experimental setup was identical to that in Ref [29], which was on a single phase
 326 austenitic stainless steel; the two materials were also tested consecutively. De-
 327 viations were not observed in the experimental lattice strains of the stainless
 328 steel and a change in diffraction sampling between the tests is highly unlikely.

329 The austenitic 304 steel which was of similar grain size tested in Ref [29]
 330 deformed solely by slip and the lattice-strain behaviour did not present any
 331 similar deviations such as those observed for the TWIP steel. Therefore, the
 332 possibility of deformation *via* slip causing the deviations is unlikely as this is
 333 not observed in the case of the 304 stainless steel. Thus it is suggested that
 334 the deviations in the lattice strain profile is due to twinning occurring in the
 335 material. An incubation plastic strain in polycrystals is often required before
 336 twinning is observed, which is conventionally attributed to either a requirement
 337 to raise the operative critical resolved shear stress for slip *e.g.* by forest hard-
 338 ening, above that required for twinning or to form a nucleating population of
 339 partial dislocations. The twinning stress is usually found to be temperature-
 340 independent. In addition, the stress in the surrounding matrix will be reduced
 341 by the twin. However, the lattice strain behaviour indicates twinning operating
 342 at low stresses and more importantly below the macroscopic yield stress. The
 343 strain relaxation associated with the deviations further suggest the initiation of
 344 twinning in the material, as once a twin has nucleated strain relaxation in the
 345 surrounding matrix is to be expected. Similar lattice strain behaviour has been
 346 observed in zirconium alloys [40, 22] and magnesium [41], where deviations in
 347 the lattice strain are present during deformation, particularly for grains in the
 348 $\{0002\}$ orientation. Among the suggested causes of these deviations, twinning
 349 has been suggested as the most plausible contributing factor.

350 Diffraction peak widths are affected by crystal size and the presence of lat-
 351 tice defects such as the accumulation or nucleation of dislocations and twins.
 352 During deformation peak broadening may occur due to many reasons. An inho-
 353 mogeneous strain field in the scattering volume, such as an increasing density
 354 of dislocations in the grains can produce peak broadening, also reducing the
 355 scattering size through twinning or having varying strains from different grains
 356 will produce peak broadening. Similarly, increasing the crystallite size, *e.g.*
 357 through twin growth, will produce an opposite effect and result in peak nar-
 358 rowing. The $\{111\}$ peak width evolution, Figure 5, indicates large variations in
 359 the peak width at low stresses, which coincide with the deviations seen in the
 360 lattice strain behaviour. It is suggested that the increase in peak width is due
 361 to twinning, which causes a reduction in the crystallite size. Similarly, as the
 362 twins grow, peak narrowing occurs as observed in Figure 5. Using the Scherrer
 363 equation and an approximation of the shape factor for a lenticular morphology

364 crystallite ($K = 1.34$), the ideal crystallite size *i.e.* twin thickness, required to
 365 produce the observed variations in the full width half maximum of the $\{111\}$
 366 peak, was calculated to be $\sim 5\text{-}10$ nm. Since the nucleation of large densities of
 367 dislocations are not expected at these low stresses it is assumed that the fluc-
 368 tuating peak widths are being influenced by twinning. The peak width begins
 369 to increase at a constant rate once yielding is fully established, *i.e.* beyond
 370 600 MPa. This steady increase may be due to an increase in the density and
 371 accumulation of dislocations post yielding. Smaller deviations *i.e.* peak narrow-
 372 ing, are subsequently observed, which also coincide with the deviations observed
 373 in the lattice strains. These may be resultant of further twin growth. Since the
 374 rate of peak broadening does not change or exhibit a plateau as deformation
 375 progresses, it can be assumed that the cessation of twinning has not occurred at
 376 22 % strain. It is suggested that the fluctuating peak width behaviour seen at
 377 lower stresses may be due to the nucleation of twins, since the twins act to reduce
 378 the effective grain size in the material, consequently this contributes to a peak
 379 broadening effect. Therefore, the constant peak broadening after ~ 600 MPa
 380 may suggest the cessation of twin nucleation and the initiation of twin thicken-
 381 ing *via* the absorption of partial dislocations. The 304 stainless steel tested in
 382 Ref [29] does not exhibit a similar behaviour to the TWIP steel; during elastic
 383 loading the $\{111\}$ peak width essentially remains constant, Figure 5.

Figure 5: $\{111\}$ peak width evolution in the tensile direction during loading of the TWIP and 304 steels.

384 4.3. Texture evolution

385 The final experimental deformation texture and the model prediction are
 386 shown in Figure 6. The weak starting texture intensified in the loading direc-
 387 tion particularly on the $\{111\}$ pole figure, but also on the $\{200\}$. The texture
 388 has also developed a distinct four-fold symmetry. Furthermore, the final tex-

ture predicted by the model shows good agreement with the experimental pole figures.

There is a pronounced fiber texture, which has developed in the $\langle 111 \rangle // \text{RD}$ and a weaker $\langle 200 \rangle // \text{RD}$ component. The final texture has four main components, the most intense being $\{110\}\langle 112 \rangle$ brass, followed by $\{110\}\langle 110 \rangle$ rotated Goss, then $\{112\}\langle 110 \rangle$ rotated copper and finally a $\{001\}\langle 100 \rangle$ cube component.

Figure 6: Final texture after deforming to 22% in the rolling direction, (a) experimental and (b) simulated (tensile direction out of page).

The evolution of the $\{111\}$ pole figure texture during deformation and the corresponding predicted texture by the model is shown in Figure 7. The texture begins to sharpen and a distinct four-fold symmetry begins to form by $\sim 7\%$ strain. Furthermore, stronger variations in intensity begin to initiate from this point. Small reorientations of the pole figures are observed at low strain *i.e.* $< 0.3\%$ strain, however these are not predicted in the simulated results. It should be noted that these reorientations predominantly occur below the macroscopic yield point of the material, therefore the model is unable to predict any slip or twin activity. The experimental observation would suggest possible twin activity or twin nucleation, which result in small reorientations in the pole figures.

The strengthening of the $\langle 111 \rangle // \text{RD}$ and the main texture components during deformation indicates that twinning activity is increasing. This would concur with evidence that twinning is well suited for the $\langle 111 \rangle // \text{RD}$ fiber component because the Schmid factor for twinning is greater than that for slip [14, 42]. Similarly the weaker $\langle 200 \rangle // \text{RD}$ fiber is better suited for slip, which is apparent in the experimental textures. The slight over prediction by the model for the intensity in the tensile axis of the $\{200\}$ pole figure, would indicate an overestimation for slip activity.

Twinning generates new orientations, which can also modify texture, therefore the texture evolution would indicate twin nucleation even at the higher strains. However, after 11.85% strain new texture components are not observed and only strengthening in intensity occurs, *i.e.* the $\{111\}$ gets bigger with fur-

Figure 7: $\{111\}$ pole figure projection of the experimental (E) and modelled (M) texture evolution during tensile loading to a final strain of 22%.

ther strain. However, some authors have suggested that the overall contribution of twinning to the deformation texture is minimal because the twins formed are very fine.

The texture index, Figure 8, indicates strengthening of the texture throughout the deformation process. A small increase in strength is detected during elastic straining, which is followed by a steady strengthening of the texture to $\sim 16\%$ strain. The rate at which strengthening occurs increases after 16% strain; the experimental pole figures show that after this strain no new texture components are formed, and further deformation only strengthens the existing texture components. Consequently this strengthening results in an increase in the texture index. The strength of the simulated textures are generally higher than the experimental observations. This is often observed in self consistent and Taylor type texture models.

The $\{111\}$ RD peak intensity evolution, Figure 9, generally exhibits an increase in the intensity during deformation. This is in contrast to the 304 austenitic steel tested in Ref [29] where intensity decreases with progressive deformation. A distinct feature of the intensity evolution is the sharp changes in intensity seen during deformation. The intensity fluctuations are even observed at nominally elastic stresses and furthermore coincide with in the lattice strain

Figure 8: Experimental and model evolution of texture index with progressive deformation.

Figure 9: Evolution of the normalised $\{111\}$ RD intensity in the tensile direction with applied stress compared to 304 stainless steel [29].

436 deviations seen in Figure 4. Abrupt changes in diffraction intensity suggest a
 437 change in the orientation of the diffracting volume, which can be caused by de-
 438 formation twinning. Therefore the sharp changes in diffraction intensity can be
 439 attributed to twinning, while the gradual increase in intensity is due to gradual
 440 grain rotation during deformation which can be attributed to slip mechanisms.

4.4. Sub-yield twinning: cyclic testing and microscopy observations

The post-deformation microstructure is shown in Figure 10; profuse twinning is observed within the grains. These can then be observed using EBSD, which shows the presence of annealing twins and also, using the band contrast, the presence of very fine twins within many of the grains, particularly those in the $\{111\}$ RD orientation, as indicated with an arrow in Figure 10(b). EBSD also highlights that whilst many of the grains are in the $\{111\}$ RD orientation, many of the grains are not; similarly the fine twins are observed only in some of these grains. Figure 11 shows low magnification (a) and high magnification (bright field) views of the twins in the post-deformed sample. Consistent with Idrissi *et al.* [43], the twins are on the order of 20 nm in thickness and are filled with faults or smaller microtwins. In addition, the matrix around the twins is relatively free of dislocations. The selected area electron diffraction (SAED) patterns indicates that two different twin systems exist, type one forming on the $(11\bar{1})$ plane and type two forming on the $(\bar{1}1\bar{1})$ plane.

In order to verify the implication from the diffraction measurements that twinning occurred below the macroscopic yield stress, some additional mechanical testing was performed. Figure 12 shows the effect of cyclic loading for 20 cycles at stresses of 200, 300 and 400 MPa, all below the macroscopic yield stress. It is observed that the sample progressively deformed, with a substantial residual strain being imparted to the sample; at 400 MPa this was $\sim 8\mu\epsilon$ per cycle. The accumulation of plastic strain during cyclic loading was also corroborated using a sample with a strain gauge mounted to rule out artefacts from the extensometer. The strain gauge also indicated an accumulation of permanent strain with each cycle.

Figure 13 shows the first unload and reload of each cycle, indicating that substantial hysteresis occurs in each case, in addition to the residual plasticity imparted. Also it can be seen that there was some relaxation that occurred during the very final stages of unloading.

Therefore even macroscopically it can be concluded that plastic deformation occurs at stresses as low as 200 MPa, less than half of the nominal 0.5% offset yield stress of 480 MPa.

Attributing these to twinning is difficult because the twin density is rather low, but thin deformation twins around 5 nm in thickness could be observed in TEM (Figure 14) after performing 1000 cycles on a sample between 10 and 200 MPa. This shows the end of a very fine twin that decomposes into the stacking faults that form the twin walls. The twin thickness observed using TEM is within the predicted range calculated using the Scherrer equation that would produce the observed sub-yield peak width broadening observed.

Therefore it can be concluded, from TEM, macroscopic testing, from the lattice strain, diffracted intensity and peak width profiles, that twinning occurs below the nominal yield stress in this material.

5. Conclusions

The texture evolution and micromechanical response of an initially untextured TWIP steel have been studied during quasi-static tensile straining along the rolling direction at 10^{-3} s^{-1} using *in-situ* synchrotron diffraction and post hoc microscopy. The following conclusions can be drawn

1. Experimental evidence from synchrotron X-ray diffraction lattice strain evolution, peak width evolution, cyclic testing using an extensometer and strain gauge coupled with TEM observations indicate that sub-yield twinning occurs.
2. Elasto-plastic self consistent modelling has been used to capture the texture and lattice strain evolution during loading.
3. The $\{111\}$ RD and $\{200\}$ RD texture evolved continuously from the very earliest stages of deformation.
4. The flow curve and texture evolution could be satisfactorily modelled using a self-consistent micromechanical model.
5. The lattice strain response show unusual deviations below macroscopic yield, a feature not observed in a similar grain size austenitic stainless steel using an identical experimental setup.
6. Corresponding deviations were also observed in the peak width and intensity; these are not felt to be artefacts as conventional linear behaviour is recovered in the later stages of deformation.
7. The very fine twins expected of a TWIP steel were observed after deformation, such that twinning can occur continuously and provide work hardening up to large strains.
8. Macroscopic cyclic testing indicated that sub-yield permanent deformation occurred even at 200 MPa, a stress less than half the macroscopic yield stress.
9. TEM examination indicated that deformation twins were induced by this cyclic testing.
10. Therefore, it is concluded that twinning can occur in the very earliest stages of deformation in the material studied, prior to the buildup of a large population of partial dislocations. However, the microscopy indicated that these twins were still associated with partial dislocations.

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Figure 10: Post deformation microstructure; (a) light micrograph and (b) EBSD map of sample deformed to 22 % strain in the rolling direction with IPF colouring relative to the RD and band contrast.

Figure 11: TEM bright field taken from the sample strained to 22 % (a) low magnification with SAED pattern of the grain while the sample is tilted onto the $[011]$ zone axis, (b) schematic of the SAED pattern shown in (a), (c) high magnification of twins and (d) microtwins within a larger twin.

Figure 12: Cyclic tensile loading using an extensometer at below macroscopic yield stresses, indicating the accumulation of strain with each loading cycle.

Figure 13: Single loading and unloading ramp indicating substantial hysteresis occurring and some relaxation.

Figure 14: TEM bright field images taken from the sample after cyclic loading (a) fine twin decomposing into stacking faults with SAED pattern taken on the $[011]$ zone axis ($g\{111\}$) and (b) ultra fine twins.