1 Micromechanics and microstructure evolution during *in situ* uniaxial
2 tensile loading of TRIP-assisted duplex stainless steels

3 Ye Tian^{a,*}, Sen Lin^a, J. Y. Peter Ko^b, Ulrich Lienert^c, Annika Borgenstam^a, Peter Hedström^a

^a*Department of Materials Science and Engineering, KTH Royal Institute of Technology,
Stockholm SE-10044, Sweden*

^b*Cornell High Energy Synchrotron Source, Cornell University, Ithaca, NY 14853, United States*

8 ^c*Deutsches Elektronen-Synchrotron, DESY Photon Science, Hamburg 22607, Germany*

9 Abstract

Two transformation-induced plasticity (TRIP) assisted duplex stainless steels, with three different stabilities of the austenite phase, were investigated by synchrotron X-ray diffraction characterization during *in situ* uniaxial tensile loading. The micromechanics and the deformation-induced martensitic transformation (DIMIT) in the bulk of the steels were investigated *in situ*. Furthermore, scanning electron microscopy supplemented the *in situ* analysis by providing information about the microstructure of annealed and deformed specimens. The dependence of deformation structure on austenite stability is similar to that of single-phase austenitic steels where shear bands and *bcc*-martensite (α') are generally observed, and blocky α' is only frequent when the austenite stability is low. These microstructural features, i.e. defect structure and deformation-induced martensite, are correlated with the micro- and macro-mechanics of the steels with elastoplastic load transfer from the weaker phases to the stronger α' , in particular this occurs close to the point of maximum rate of α' formation. A clear strain-hardening effect from α' is seen in the most unstable austenite leading to a pronounced TRIP effect.

Keywords: synchrotron diffraction, *in situ* tensile loading, duplex stainless steel, load partitioning, TRIP-assisted steel, micromechanics.

26

27

1.Introduction

Duplex stainless steels constitute of a mixture of approximately equal amounts of the austenite and ferrite (hereinafter referred to as γ and α , respectively). They have good corrosion resistance and mechanical properties with, in general, yield strength superior, but ductility inferior, to austenitic grades. The duplex stainless steels find frequent use in storage containers and piping, especially in harsh environments and in conditions where austenitic grades would be susceptible to stress corrosion [1]. Recently, there has been a development effort aimed at increasing the ductility and formability of the duplex stainless steels. In designing the alloy, the transformation-induced plasticity (TRIP) phenomenon has been utilized by tuning the γ stability via suitable alloying additions. The TRIP effect is achieved by deformation-induced martensitic transformation (DIMP), providing additional work hardening that postpones plastic instability and necking. The TRIP effect is successfully utilized in numerous steels, not only in metastable austenitic stainless steels, but also in TRIP carbon steels [2], quenching & partitioning steels [3], maraging steels [4] and other high strength steels [5–7]. The new TRIP-assisted duplex stainless steels, hereinafter referred to as TDSS, can have total elongations over ~60% and ultimate tensile strengths up to ~1 GPa [8].

The deformation behaviour of the conventional duplex stainless steels is complex and the interplay between γ and α has been studied extensively using *in situ* X-ray and neutron diffraction experiments [9–13]. The inhomogeneous deformation behaviour with load partitioning between the phases [9] and textural components [12] has been documented. The TDSS are, however, even more complex due to the possible transformation of the metastable austenite to two kinds of products, i.e. *hcp*- and *bcc*-martensite (hereinafter referred to as ϵ and α' , respectively), during deformation. Many previous studies on DIMP have been devoted to the investigation of TRIP carbon steels [2,4,14–16] and metastable austenitic stainless steels [17–20], but studies of DIMP in TDSS are rarely presented, apart from some recent reports [8,21–23].

In order to tailor the mechanical properties of TDSS, a thorough understanding of the micromechanics and the TRIP effect during deformation is required. The excellent combination

of strength and ductility of TRIP-assisted multiphase steels can be attributed to the defect interaction, phase and texture evolution, composite strengthening [22,24], etc. These can be largely influenced by the γ stability [15,24–26], which in turn can be represented by the stacking fault energy (SFE) [17]. However, the available reports of TDSS have mainly focused on the DIMT itself [21–23,27] and a more complete view of the deformation behaviour of TDSS with different γ stability is lacking.

The aim of the present work is to study the deformation behaviour of two TDSS with different γ stability and, furthermore, to investigate the effect of increasing temperature on deformation in one of the steels. High-energy X-ray diffraction (HEXRD) measurements during *in situ* uniaxial tensile loading are applied to study the micromechanics and DIMT. It is further supplemented by electron microscopy investigations.

2. Experimental methods

2.1 Materials

The experiments were carried out on two TDSS, FDX27 and FDX25, supplied by Outokumpu Stainless. The as-received state was 1 mm thick steel sheets with the chemical compositions given in Table 1. Both steels were tested at room temperature (RT) and, FDX25 was also tested at 45 °C. The testing conditions are hereinafter referred to as FDX27@RT, FDX25@RT and FDX25@45. Dog-bone-shaped tensile specimens with a gauge length of 3 mm and gauge width of 1 mm, shown in the upper left corner of Fig. 1, were cut from the steel sheets using electrical discharge machining. The specimens were then ground and polished to a final thickness of 0.8 mm. In the grip section of the specimens, two holes were drilled for pin-hole attachment in the tensile rig.

Table 1 Chemical compositions of the investigated steels (wt.%).

	C	Si	Mn	Cr	Ni	Mo	Ti	Al
FDX25	0.023	0.38	2.52	20.26	1.48	0.4	0.001	0.009
FDX27	0.021	0.38	0.93	19.97	3.05	1.15	0.003	0.02
	Nb	Cu	Co	N	W	V	Fe	
FDX25	0.004	0.5	0.04	0.22	<0.01	0.06	Bal.	
FDX27	0.004	0.32	0.06	0.18	0.01	0.06	Bal.	

2.2 *In situ* loading experiments

2.2.1 High-energy X-ray diffraction measurements during *in situ* tensile loading

In situ uniaxial tensile loading experiments were performed in a customized load frame, called rotational and axial motion system (RAMS) dedicated for HEXRD experiments, which is shown in Fig. 1. A detailed description of the RAMS can be found in Shade *et al.* [28]. During tensile tests at 45 °C the temperature was regulated by a furnace containing halogen bulbs and an elliptical mirror to focus the light onto the specimen. Two thermocouples were used for furnace control and two for specimen temperature measurements. The load was applied along the rolling direction of the sheet steel, and was measured using a load cell with a 2 kN capacity. Interrupted tensile loading was performed using displacement control at a strain rate of 10^{-4} s^{-1} up to an applied true strain of 0.26 (evaluated via the displacement of the load frame). HEXRD measurements were performed *in situ* during loading. The experiments were conducted at the F2 beamline at the Cornell High Energy Synchrotron Source (CHESS), U.S, using an X-ray beam with an energy of 61.332 keV (0.20218 Å), collimated to a size of $0.8 \times 2 \text{ mm}^2$ (V $\times$ H). An area detector (GE Detector 2048 $\times$ 2048 pixels, $200 \times 200 \text{ }\mu\text{m}^2$ per pixel) was placed about 1012 mm behind the specimen to collect the diffraction patterns. For each strain step, a 2D diffraction pattern was acquired for a constant ω angle of 0° (incoming beam parallel to specimen's normal direction). The 2-theta (2 θ) angle is in the radial direction on the detector. Prior to the *in situ* experiments a standard CeO₂ powder specimen was measured for calibration purposes.

Fig. 1 Schematic illustration of the HEXRD setup for the *in situ* loading experiments. (The furnace is movable and can be translated in and out of the X-ray beam path by a slide rail.)

2.2.2 Data analysis for phase quantification

The 2D diffraction data was calibrated and processed using mainly the GSAS-II software [29], and the patterns acquired during the *in situ* experiments were integrated over 360° in the azimuthal direction (η). Fitting of the peaks was performed by a combination of single-peak and multiple-peak fitting. This procedure enabled good fitting for all peaks, even for low intensity peaks from the ϵ phase. Fig. 2a shows an example of the raw 2D pattern, Fig. 2b shows the overview of integrated 1D patterns and Fig. 2c shows the least-squares fitting of peaks using pseudo-Voigt functions.

Fig. 2 Illustration of the data processing procedure for phase quantification, (a) 2D diffraction pattern of FDX27@RT at 0.26 true strain; (b) 1D diffraction profiles for different true strains of FDX27@RT; (c) Fitting result for the indicated area of the 1D profile in (b), i.e. FDX27@RT deformed at 0.26 true strain.

The direct comparison method [30] was used to determine the volume fractions of the phases. This methodology is intended for powder patterns, assuming a random texture, but it is also known that by measuring multiple orientations and peaks for each phase the effect of texture on phase quantification will be minimized [31]. The volume fraction is evaluated using:

$$V_i = (\frac{1}{n} \sum_{j=1}^n I_i^j / R_i^j) / (\frac{1}{p} \sum_{k=1}^p I_\gamma^k / R_\gamma^k + \frac{1}{q} \sum_{l=1}^q I_\varepsilon^l / R_\varepsilon^l + \frac{1}{r} \sum_{m=1}^r I_{bcc}^m / R_{bcc}^m) \quad (1)$$

where V_i is the volume fraction of phase- i ; $I_i^j (I_i^{hkl})$ is the integrated intensity for the $\{hkl\}$ plane of phase- i ; $R_i^j (R_i^{hkl})$ is the scattering factor which can be expressed as:

$$R_i^j = v^{-2} |F|^2 p (1 + \cos^2 2\theta) / (\sin^2 \theta \cos \theta) e^{-2M} \quad (2)$$

where v represents the volume of the unit cell; F is the structure factor of plane hkl , p is the multiplicity factor; θ is the Bragg angle, $(1 + \cos^2 2\theta) / (\sin^2 \theta \cos \theta)$ is the Lorentz-polarization factor, and e^{-2M} is the Debye-Waller factor [30].

2.2.3 Data analysis for strain/stress analysis

The elastic lattice strains along the loading direction (integration over $5^\circ \eta$ sectors) were evaluated using the following equation:

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0} \quad (3)$$

where d_{hkl} represents the measured lattice spacing at a given deformation step, and d_{hkl}^0 is the strain-free lattice spacing. To evaluate the hkl -specific lattice strains in this study, d_{hkl}^0 was assumed to be the azimuthally averaged d_{hkl} after the specimen had been mounted on the load frame and when a stress of between 20-60 MPa was applied to fixate the specimen position. This means that the absolute hkl -specific lattice strains are uncertain due to possible inaccuracies in d_{hkl}^0 and we will only discuss the relative change of ε_{hkl} , i.e. $\Delta \varepsilon_{hkl}$.

For the determination of the stress partitioning to a specific phase (σ_{phase}), accurate d_{hkl} values and their shifts (d_{hkl} -shift) are also required [32], and therefore an alternative methodology was applied. The software MAUD and Rietveld analysis were used, and the strain-free lattice parameter was refined during the analysis. Furthermore, using built-in models in MAUD, the total stress in constituent phases, root mean square (r.m.s.) microstrain, the d_{hkl} -shift due to the

formation of stacking fault, texture etc. were considered [33,34]. The bulk-path geometric mean (BPGeo) micro-mechanical model [35] was used to predict σ_{phase} . The BPGeo model is practically equal to a self-consistent model [36], but it is simpler for bulk materials. The elastic stiffness coefficients used as input for the model are presented in Table 2. The imposed stress condition for the uniaxial tensile loading can be regarded as $\sigma_1 = \sigma_2 = 0$, $\tau_{ij} = 0$ ($i \neq j$), $\sigma_3 = \sigma$ (loading direction). The Warren model was applied to deal with the d_{hkl} -shift caused by the formation of stacking faults in γ [37]. Arbitrary texture was used for the Rietveld refinement, and no orientation distribution function weighted averaging procedures were applied for the assessment of σ_{phase} . The r.m.s. microstrain model developed by Popa [38] was used to describe the peak broadening due to microstrain and crystallite size [39]. A more detailed description of MAUD and the applied models in MAUD can be found in Refs. [33,34]. Fig. 3 shows the measured (bottom) and fitted (top) diffraction patterns as a function of η for FDX27@RT at 0.26 true strain.

Table 2 The stiffness of FDX25 and FDX27 [40].

	C_{11} (GPa)	C_{12} (GPa)	C_{44} (GPa)
Ferrite	231.4	134.7	116.4
Austenite	197.5	124.5	122.0

Fig. 3 2D representation of the diffraction data with 2θ versus η and the colour indicating the intensity of diffraction spots from black to red, FDX27@RT at 0.26 true strain. (Top: Fitted results. Bottom: Measured results)

2.3 Electron microscopy and estimation of austenite stacking fault energy

The microstructure of the specimens were characterized before and after tensile loading using electron channelling contrast imaging (ECCI) [41] and electron backscatter diffraction (EBSD) in a field-emission scanning electron microscope (FE-SEM) JEOL 7800F. In order to avoid introducing deformation-induced martensite by mechanical polishing, all specimens were prepared using electrolytic polishing for about 30 s after polishing with fine grade sand paper. The electrolyte was a mixture of perchloric acid and acetic acid in 1:9 proportions. Post-processing of EBSD data was performed using the MTEX software [42]. Automatic filtering was applied to separate the two *bcc* phases, α and deformation-induced α' . The main filters applied were band contrast, grain size and grain orientation spread (GOS) [43]. However, this was not sufficient to separate the *bcc* phases and it was therefore complemented with manual selection based on length-to-width aspect ratio. In the end, the *bcc* phases were successfully distinguished from each other.

To be able to estimate the stability of the γ , the chemical compositions of γ and α were analysed using energy dispersive X-ray spectroscopy in a transmission electron microscope (TEM-EDS) JEOL JEM-2100F. The thin-foil specimens were prepared using focused ion beam and a dual-beam instrument FEI Nova 600. The SFEs can be estimated using thermodynamic modelling by calculating the molar Gibbs free energy difference between γ and ϵ ($\Delta G^{fcc \rightarrow hcp}$) and adding the contributions of the interfacial energy of the γ/ϵ interface σ' [18,44,45] and the strain energy (E^{strain}):

$$\gamma_{SF} = 2\rho(\Delta G^{fcc \rightarrow hcp} + E^{strain}) + 2\sigma' \quad (4)$$

where ρ is the density of atoms along the $\{111\}\gamma$ planes [46]. Both σ' and E^{strain} are difficult to evaluate; however, by assuming that these two parameters are the same for all three conditions, we can directly calculate $\Delta G^{fcc \rightarrow hcp}$ and rank the conditions according to increasing SFE. The Thermo-Calc [47] software system with database TCFE8 [48] were used to calculate $\Delta G^{fcc \rightarrow hcp}$.

3. Results

3.1 Thermodynamic estimation of γ stability

The chemical compositions of γ and α measured using TEM-EDS could only account for the major heavier elements (Si, Mn, Cr, Ni, Mo), and therefore, other elements were evaluated using Thermo-Calc equilibrium calculations at the annealing temperature (1050 °C). The results are listed in Table 3. The $\Delta G^{fcc \rightarrow hcp}$ values of the three conditions were then calculated to be -166, 119 and 155 J mol⁻¹ for FDX27@RT, FDX25@RT and FDX25@45 respectively. Thus, the γ stability, according to the SFE, was ranked from low to high in the order: FDX27@RT, FDX25@RT and FDX25@45. In addition, the $\Delta G^{fcc \rightarrow bcc}$ was also calculated to be -2572, -2258 and -2159 J mol⁻¹ for FDX27@RT, FDX25@RT and FDX25@45 respectively. Hence, the order of stability of γ for the alloy conditions is estimated to be the same with respect to either ϵ or α' transformation. The absolute values of $\Delta G^{fcc \rightarrow bcc}$, however, are uncertain.

Table 3 Chemical compositions (wt. %) of α and γ obtained from TEM-EDS and Thermo-Calc for FDX25 and FDX27.

	Fe	Si	Mn	Cr	Ni	Mo	Cu*	C*	N*	Co*	W*	V*	Al*	Ti*	Nb*
FDX25(γ)	Bal.	0.3	2.7	20.1	1.7	0.4	0.55	0.03	0.29	0.04	0.008	0.05	0.008	0.00001	0.002
FDX27(γ)	Bal.	0.3	1.0	19.8	3.2	0.9	0.35	0.03	0.24	0.07	0.008	0.05	0.02	0.00001	0.002
FDX25(α)	Bal.	0.4	2.3	23.2	0.9	0.5	0.40	0.01	0.04	0.03	0.02	0.06	0.01	0.00002	0.004
FDX27(α)	Bal.	0.4	0.8	22.9	1.7	1.5	0.25	0.01	0.03	0.05	0.01	0.07	0.03	0.00002	0.005

* represents the value calculated by Thermo-Calc.

3.2 Stress-strain behaviour and deformation-induced martensitic transformation

True stress-strain and work hardening rate curves for the three conditions are shown in Fig. 4. The work hardening behaviours for the three conditions were significantly different above about 0.2 true strain (inset of Fig. 4). FDX27@RT has the highest work hardening rate above 0.2 true strain, while FDX25@45 has the lowest. In addition, the work hardening rate curves for the three conditions reach the minimum values at different true strains; FDX27@RT reaches its minima at around 0.16, FDX25@RT at around 0.2, whereas FDX25@45 has an approximately constant work hardening rate after 0.22.

Fig. 4 True stress-strain curves and work hardening rate curves of FDX27@RT, FDX25@RT and FDX25@45.

All steels investigated in the present work contain two phases, α and γ , in the annealed state. Upon loading the intensities of the α/α' peaks increase while the intensities of the γ peaks decrease (Fig. 2b). A weak peak for the 100ϵ was also distinguished (Fig. 2c), as exemplified for FDX27@RT and FDX25@RT after 0.05 true strain in Fig. 5a and b, respectively. The evaluated volume fractions of different phases are presented in Fig. 5. All three conditions have the same trend, increasing strain leads to an increasing fraction of α' . At the same strain, more α' forms in FDX27@RT than in the two FDX25 conditions, and the least α' was observed in FDX25@45. ϵ was detected in both FDX27@RT and FDX25@RT but not in FDX25@45. It is also indicated that the fraction of ϵ decreases with increasing formation of α' in FDX27@RT, see Fig. 5b₁ and b₂. It should be noted that the fraction of ϵ is very low and small errors in peak fitting and grain rotation could also affect the results. Hence, the absolute quantification of ϵ volume fraction was not considered fully reliable due to the influence of texture when only one peak is considered [31].

Fig. 5 Volume fraction evolution of γ , ε and α' against true strain of (a) FDX27@RT, (c) FDX25@RT and (d) FDX25@45 conditions. (error bars are small and covered by the symbol) (b) 2D maps of diffraction profiles as a function of η for FDX27@RT at two different true strains. Note: Quantification of ε is not considered fully quantitative due to texture, but the trend should be accurate.

3.3 Load partitioning between hkl-specific orientation families and phases

Lattice strain evolution during uniaxial tensile loading along loading direction for the three conditions are shown in Fig. 6. In the elastic region (below ~500MPa true stress), almost linear responses of lattice strains in terms of applied stress are observed for all studied {hkl} planes. The different slopes of these lines are associated with the cubic elastic anisotropy factor $A_{hkl} = (h^2k^2 + k^2l^2 + l^2h^2)/(h^2 + k^2 + l^2)^2$ [26,49], and larger A_{hkl} indicates greater stiffness. Parallel to the loading direction, the softest hkl-orientation is observed to be {200} for both γ and α phases which is expected. The stiffest hkl-orientation for γ is {111} for all conditions as expected; and the stiffest hkl-orientations for α are {211} for the FDX27@RT and {110} for the FDX25 conditions. However, their small differences cannot be distinguished confidently due to some measurement uncertainties in the elastic deformation regime.

Fig. 6 The evolution of the elastic lattice strains for γ and α phases in (a) FDX27@RT, (b) FDX25@RT and (c) FDX25@45. (error bars are small and covered by the symbols)

Two main load redistributions are visualized by the deviations from linear evolution of the lattice strains in the elastoplastic and plastic regimes for the three conditions. The first load redistribution occurs around the yield point. For instance at about 600 MPa true stress, a sudden drop of lattice strain in $\{220\}\gamma$ plane and a slower increase of lattice strain in $\{111\}\gamma$ plane are observed in FDX27@RT (black circle in Fig. 6a). The deviation from linearity indicates the

start of plastic deformation in these hkl-orientation families of γ . In contrast, grains with their $\langle 200 \rangle$ direction parallel to the loading direction ($\langle 200 \rangle$ -oriented) remain almost elastic indicated by the faster increase of lattice strain in $\{200\}\gamma$ plane. Further loading to about 650MPa enables the plastic deformation in $\langle 200 \rangle$ -oriented grains as indicated by the slower increase of lattice strain. Similar behaviours for γ are also observed for the two other conditions close to this load range as shown in Fig. 6b, c.

The start of the α' formation for each condition is indicated by α' -black lines in Fig. 6, but the other major load redistribution observation is not seen until the largest transformation rate of α' (LTR-black lines). The lattice strain curves all deviate from linearity as the plastic deformation increases. A zig-zag behaviour is seen for the $\{111\}\gamma$ and $\{220\}\gamma$ lattice planes, especially after the initial formation of α' . This behaviour is more obvious in FDX27@RT and FDX25@RT, where extensive γ has transformed. It should be noticed that larger errors of the $\{220\}\gamma$ lattice strain exist due to the low peak intensity for the last few measurement points.

As presented in section 2.2.3, the stress distribution of specific phases were obtained from the Rietveld analysis. The stress-strain curves for γ , α and the bulk are illustrated in Fig. 7. The γ experienced higher stress than α for the two FDX25 conditions, while slightly lower than α in FDX27@RT at similar true strains. The stress in α was only evaluated until the initial formation of α' , and in that strain range, the load partitioning between γ and α remains similar. The stress of γ at about the yield point (0.05 true strain) shows an increasing trend with the increasing γ stability as indicated by black arrows. At the LTR and onwards, the load starts to be transferred from the γ to the *bcc* phase, most probably the stronger α' . This is seen most clearly in FDX27@RT.

Fig. 7 The partitioning of stress to the constituent phases and the applied stress versus applied true strain in TDSS subjected to a uniaxial tensile stress. (a) FDX27@RT, (b) FDX25@RT and (c) FDX25@45. (error bars are small and covered by the symbols)

3.4 Microstructure

The as-received microstructures of both steels are displayed in Fig. 8a and d. They are characterized by the typical duplex appearance with a lamellar structure consisting of alternating α and γ regions, indicated by blue and red, respectively. The phase fractions in the as-received state, evaluated by EBSD, are 42 vol.% α and 58 vol.% γ in FDX25, 35.6 vol.% α and 64.4 vol.% γ in FDX27, which are in agreement with the HEXRD results: 40.2 vol.% α and 59.8 vol.% γ in FDX25 and 38.3 vol.% α and 61.7 vol.% γ in FDX27.

The grain size of α differs between the two steels and is much smaller in FDX27 than that in FDX25, 3.5 μm and 7.9 μm respectively. The grain size of γ is more similar between the two alloys, 2.9 μm and 3.7 μm respectively.

Fig. 8 EBSD phase maps (PM) and GOS maps of the investigated alloys in the annealed state: (a-c) FDX25, (d-f) FDX27. (α and γ are indicated by blue and red, respectively in (a) and (b); the misorientation levels are presented according to the colour scale bar to the right.)

The GOS analysis could be related to strain levels and is not that sensitive to grain size and step size as compared to the kernel average misorientation analysis, according to Shen [50]. From GOS maps, γ phase of the two steels reveals rather small deformation at 0 true strain (Fig. 8c and f). The GOS map of α for FDX25 indicates low level of strain as well (Fig. 8b), but much higher values are found for FDX27 (Fig. 8e) indicating the existence of pre-strain in the α for FDX27 in the as-received state.

The deformation microstructure of the three conditions after tensile loading to 0.26 true strain is shown in Fig. 9. The deformation microstructures of α and γ are quite different. In general, shear bands form extensively in γ intersecting with each other, while α is mainly characterized by dislocation cells similar to the deformation in conventional duplex stainless steels. However, the deformation microstructures of γ were observed to be quite different depending on the γ stability in TDSS. For the least stable condition (FDX27@RT), most of the γ grains were observed to have fully transformed to blocky α' as shown in Fig. 9a. The enlarged area of this image indicates the formation of blocky α' . This type of blocky α' originates from defects, especially shear bands and grain boundaries, and grow into the γ which is rather unstable. Small units of α' were also observed within individual shear bands. For FDX25@RT, most of the γ grains deformed through shear bands as shown in Fig. 9b. α' was observed to mainly form at the intersections of shear bands in FDX25@RT as shown in the enlarged area of Fig. 9b, while the intersections may not be the necessary nucleation sites for α' in FDX27@RT. Fig. 9c shows the deformation microstructure of FDX25@45 which has the most stable γ , and it is observed that most of the γ grains deformed via shear bands. Some grains could even be characterized by dislocation cells as indicated by yellow arrows in Fig. 9c. The enlarged area of this image shows that α' hardly forms even with extensive deformation and intersecting shear bands.

Fig. 9 ECCI images of deformation microstructure for specimens tensile loaded to 0.26 true strain, (a) FDX27@RT; (b) FDX25@RT; (c) FDX25@45. SBs: Shear bands, DCs: Dislocation cells.

Fig. 10 shows the phase maps of all conditions at 0.26 true strain. The phase fraction trend of γ and α' from EBSD is in agreement with HEXRD results, but ϵ could not be characterized by EBSD. Fig. 10a, c and e shows the phase maps of γ , α and α' , and Fig. 10b, d and f shows the phase maps of γ and α' after filtering out α .

Fig. 10 EBSD phase maps of all conditions at 0.26 true strain, (a, b) FDX27@RT; (c, d) FDX25@ RT; (e, f) FDX25@45. (α/α' and γ are indicated by blue and red, respectively.)

4. Discussion

4.1 Deformation behaviour of individual constituents

In stable duplex stainless steels, it is well-known that the deformation behaviour of the γ and α phases depends on the specific composition. In particular, N is known to have a large effect. Assuming both phases have the same grain size, the γ phase will be the weaker phase if the N content is below 0.12 wt.%; whereas if the N content is above 0.12 wt.%, α will be the weaker phase [10]. The reason is that N is effective in increasing the SFE of γ and thus also the flow strength. In the present work, both steels have a rather high N content and the load partitioning between γ and α is somewhat different. For the FDX25 steels, the γ is the stronger phase at both temperatures investigated, and in addition to the high N content this should also be attributed to the significantly smaller grain size of the γ phase. For the FDX27 alloy, the γ is the weaker phase and both γ and α experience stresses close to the applied stress initially as shown in Fig. 7a. In this case the γ and α grain sizes are more similar and, moreover, the α phase is plastically deformed already in the as-received state. Hence, both Hall-Petch hardening and cold-work hardening is suggested to be responsible for elevating the strength of the α phase to be comparable with the γ .

When looking into the deformation of the microstructure, it was found that the α phase developed the characteristic dislocation cell substructure as shown in Fig. 9. This substructure develops due to the relative ease of cross-slip in the *bcc* phase (24 potential slip systems, $\{110\}\langle 111\rangle$ and $\{112\}\langle 111\rangle$) [8,51]. The deformation of the γ phase on the other hand is more dependent on the alloy composition and the deformation temperature. Based on the thermodynamic estimations, the γ stability can be ranked as FDX27@RT, FDX25@RT, FDX25@45 from lower to higher, which will affect the deformation microstructure. For lower γ stability (FDX27@RT and FDX25@RT), the γ deformed via shear bands, and α' formed frequently as shown in the enlarged area of Fig. 9a, b. The α' nucleates at shear bands preferentially adjacent to ϵ (enlarged area of Fig. 9a and b), where a lower activation energy is needed for the formation of α' [25]. This is in agreement with our previous observations on single-phase austenitic alloys [52]. After extensive deformation, much of the γ has then transformed to α' (Fig. 10b, d). The transition from shear bands (including ϵ) to α' is further indicated by the slight decrease of the ϵ fraction at higher applied plastic strains as shown in Fig. 5b₁₋₂ due to the consumption of ϵ after LTR. As reported for single-phase austenitic alloys, the intersections of shear bands should be the preferred nucleation sites for α' [53,54]. This is also the dominant feature for the more stable γ in the FDX25@RT where the α' mainly formed at the intersections of shear bands, as shown in the enlarged area of Fig. 9b. When the γ stability is even higher as in FDX25@45, both shear bands and dislocation cells were observed as shown in Fig. 9c. The volume fraction of α' is the lowest for FDX25@45 and slightly higher for FDX25@RT. In FDX27@RT, the most α' forms upon deformation and it is also found that for the most unstable γ , the formation of α' units is also frequent at individual shear bands as shown in the enlarged area of Fig. 9a. This observation as well as the existence of blocky α' indicate that the deformation behaviour of unstable γ in the TDSS resemble that of unstable γ in metastable single-phase austenitic alloys [25].

Considering the ϵ phase in TDSS, it appears in significantly lower fractions as compared to our previous investigations of single-phase austenitic alloys but with similar tendency for α' formation [25]. This could be attributed to the much lower driving force for γ to ϵ and

comparable driving force for γ to α' for TDSS at RT compared with single-phase austenitic alloys [52]. But it should also be kept in mind that the calculations of driving force for multicomponent alloys can be uncertain and here we therefor only consider the relative order between the alloys. In the FDX25@45, the direct formation of α' without any ϵ formation was observed. This is the steel with the lowest α' fraction but still about 10%. Hence, it indicates that α' can form at intersections of shear bands, twin boundaries and dislocation walls [18,55] without ϵ . This could be due to that α' can form at less potent sites with the additional help from piled-up dislocations [56].

4.2 Load partitioning and work hardening

In the stable duplex stainless steels, it is known that residual stresses partition between γ and α after solution treatment and quenching. γ is generally put in tension whereas α is in compression due to their different thermal expansion coefficients [10]. The residual stresses are not further investigated in the present work, instead the focus is on the load partitioning during tensile loading. Starting with the load partitioning between the two phases at the yield point, γ is, as mentioned, the stronger phase bearing more load in the FDX25 conditions, whereas α is stronger in FDX27@RT. It is noteworthy that the critical resolved shear stress for yielding of γ changes with the stability of γ as indicated by the black arrows in Fig. 7. This could be explained by the stress dependence of stacking fault separation in low SFE materials [51].

At higher strains with increasing work hardening, we can see in Fig. 7 that the load in γ is continuously transferred to other constituent phases. Since the formation of α' is responsible for the increasing work hardening [17], this clearly indicates that more load is transferred to the α' phase at this stage. At high strains, it can also be seen that the *bcc* phases form a percolating network (Fig. 10a, c and e), especially for the FDX27@RT, which has been described as critical for the effective load transferring to the α' phase in single-phase austenitic alloys [57,58]. In the FDX27@RT, this leads to a dramatic work hardening at high plastic strains with a large fraction of α' (Fig. 7a), indicating a strong TRIP effect.

Focusing attention towards the load partitioning between differently oriented grain families, there are two distinct load redistribution stages as shown in Fig. 6. The first load redistribution occurs around the yield point. For instance, in the FDX27@RT, the plastic deformation starts in γ and the shear bands in $\langle 220 \rangle$ - and $\langle 111 \rangle$ -oriented γ grains are firstly activated when the critical resolved shear stress is achieved. The load is, therefore, transferred to γ grains with another orientation and α grains which are still deformed elastically. Further increase of the applied stress gives rise to the continuous activation of new γ grains and even α grains until all grains are deformed plastically. On the other hand, in FDX25 conditions, the plastic deformation starts from α causing a delayed plastic deformation in γ grains. The second load redistribution occurs around the LTR as seen from the deviation from linearity of the lattice strain curves, especially the following zig-zag behaviour for $\{111\}\gamma$ and $\{220\}\gamma$ lattice planes. Similar to other multiphase materials, the load could be transferred when a new phase forms or the grains rotate [26]. Hence, the relaxation of γ occurs when the load is continuously transferred towards the stronger α' .

5. Conclusions

- (1) The mechanical properties of TDSS, especially the work hardening behaviour, depend on the stability of γ and it is influenced by the load partitioning and redistribution between phases and differently orientated grains.
- (2) γ deforms in a similar manner as in metastable austenitic alloys but with less formation of ϵ . This is associated with a difference in SFE of γ . The deformation microstructure of α is mainly characterized by dislocation cells.
- (3) Both transformation sequences, $\gamma \rightarrow \epsilon \rightarrow \alpha'$ and $\gamma \rightarrow \alpha'$, have been observed. ϵ can reduce the activation energy for α' formation, while more severe deformation is required for α' formation without ϵ .
- (4) The work hardening behaviour is largely associated with the formation of α' martensite. After extensive deformation, it is indicated that the coalescence of *bcc* phases, α and α' , gives rise to percolating clusters increasing the work hardening.

(5) During tensile loading, the initial load partitioning depends on the elastic properties of the phases and orientations. The first load redistribution occurs due to the elastic and plastic anisotropy and the second load redistribution takes place at the point of the highest α' transformation rate.

Acknowledgement

This work was performed within the VINN Excellence Centre Hero-m 2i, financed by VINNOVA, the Swedish Government Agency of Innovation Systems, Swedish Industry and KTH. This work is based upon research conducted at the Cornell High Energy Synchrotron Source (CHESS) which is supported by the United States National Science Foundation under award DMR-1332208. The authors are grateful to Darren Dale, Margaret Koker, Prasath Babu R, Yan Ma, Fei Huyan and Basil Blank for technical assistance, and Jan Y. Jonsson from Outokumpu for providing the materials. Y. Tian would like to express his gratitude to the support from CSC (China Scholarship Council) and Axel Hultgrens foundation. P. Hedström acknowledges the support from Jernkontoret (Swedish Steel Producers' association).

References

- [1] H. Farnoush, A. Momeni, K. Dehghani, J.A. Mohandesi, H. Keshmiri, Hot deformation characteristics of 2205 duplex stainless steel based on the behavior of constituent phases, *Mater. Des.* 31 (2010) 220–226.
- [2] R. Blondé, E. Jimenez-Melero, L. Zhao, J.P. Wright, E. Brück, S. van der Zwaag, N.H. van Dijk, High-energy X-ray diffraction study on the temperature-dependent mechanical stability of retained austenite in low-alloyed TRIP steels, *Acta Mater.* 60 (2012) 565–577.
- [3] X. Tan, Y. Xu, X. Yang, Z. Liu, D. Wu, Effect of partitioning procedure on microstructure and mechanical properties of a hot-rolled directly quenched and partitioned steel, *Mater. Sci. Eng. A.* 594 (2014) 149–160.
- [4] M.-M. Wang, C.C. Tasan, D. Ponge, a. Kostka, D. Raabe, Smaller is less stable: Size effects on twinning vs. transformation of reverted austenite in TRIP-maraging steels, *Acta Mater.* 79 (2014) 268–281.
- [5] F.G. Caballero, M.J. Santofimia, C. García-Mateo, J. Chao, C.G. de Andrés, Theoretical design and advanced microstructure in super high strength steels, *Mater. Des.* 30 (2009) 2077–2083.
- [6] H. Luo, H. Dong, M. Huang, Effect of intercritical annealing on the Lüders strains of medium Mn transformation-induced plasticity steels, *Mater. Des.* 83 (2015) 42–48.

- 523 [7] Y. Zhao, L. Le Joncour, A. Baczmański, E. Gadalińska, S. Wroński, B. Panicaud, M.
524 François, C. Braham, T. Buslaps, Stress distribution correlated with damage in duplex
525 stainless steel studied by synchrotron diffraction during plastic necking, *Mater. Des.*
526 113 (2017) 157–168.
- 527 [8] C. Herrera, D. Ponge, D. Raabe, Design of a novel Mn-based 1GPa duplex stainless
528 TRIP steel with 60% ductility by a reduction of austenite stability, *Acta Mater.* 59
529 (2011) 4653–4664.
- 530 [9] J. Johansson, M. Odén, X.-H. Zeng, Evolution of the residual stress state in a duplex
531 stainless steel during loading, *Acta Mater.* 47 (1999) 2669–2684.
- 532 [10] J.J. Moverare, M. Odén, Influence of elastic and plastic anisotropy on the flow
533 behavior in a duplex stainless steel, *Metall. Mater. Trans. A.* 33 (2002) 57–71.
- 534 [11] P. Hedström, T.S. Han, U. Lienert, J. Almer, M. Odén, Load partitioning between
535 single bulk grains in a two-phase duplex stainless steel during tensile loading, *Acta*
536 *Mater.* 58 (2010) 734–744.
- 537 [12] N. Jia, R.L. Peng, Y.D. Wang, G.C. Chai, S. Johansson, G. Wang, P.K. Liaw,
538 Interactions between the phase stress and the grain-orientation-dependent stress in
539 duplex stainless steel during deformation, *Acta Mater.* 54 (2006) 3907–3916.
- 540 [13] M. Eskandari, a. Najafizadeh, a. Kermanpur, Effect of strain-induced martensite on
541 the formation of nanocrystalline 316L stainless steel after cold rolling and annealing,
542 *Mater. Sci. Eng. A.* 519 (2009) 46–50.
- 543 [14] E. Jimenez-Melero, N.H. van Dijk, L. Zhao, J. Sietsma, S.E. Offerman, J.P. Wright, S.
544 van der Zwaag, The effect of aluminium and phosphorus on the stability of individual
545 austenite grains in TRIP steels, *Acta Mater.* 57 (2009) 533–543.
- 546 [15] F. Lani, Q. Furnémont, T. Van Rompaey, F. Delannay, P.J. Jacques, T. Pardoen,
547 Multiscale mechanics of TRIP-assisted multiphase steels: II. Micromechanical
548 modelling, *Acta Mater.* 55 (2007) 3695–3705.
- 549 [16] E. Cakmak, H. Choo, K. An, Y. Ren, A synchrotron X-ray diffraction study on the
550 phase transformation kinetics and texture evolution of a TRIP steel subjected to
551 torsional loading, *Acta Mater.* 60 (2012) 6703–6713.
- 552 [17] J. Talonen, H. Hänninen, Formation of shear bands and strain-induced martensite
553 during plastic deformation of metastable austenitic stainless steels, *Acta Mater.* 55
554 (2007) 6108–6118.
- 555 [18] T. Masumura, N. Nakada, T. Tsuchiyama, S. Takaki, T. Koyano, K. Adachi, The
556 difference in thermal and mechanical stabilities of austenite between carbon- and
557 nitrogen-added metastable austenitic stainless steels, *Acta Mater.* 84 (2015) 330–338.
- 558 [19] N. Li, Y.D. Wang, W.J. Liu, Z.N. An, J.P. Liu, R. Su, J. Li, P.K. Liaw, In situ X-ray
559 microdiffraction study of deformation-induced phase transformation in 304 austenitic
560 stainless steel, *Acta Mater.* 64 (2014) 12–23.
- 561 [20] Y.B. Das, A.N. Forsey, T.H. Simm, K.M. Perkins, M.E. Fitzpatrick, S. Gungor, R.J.
562 Moat, In situ observation of strain and phase transformation in plastically deformed
563 301 austenitic stainless steel, *Mater. Des.* 112 (2016) 107–116.
- 564 [21] Y. Li, W. Li, J.C. Hu, H.M. Song, X.J. Jin, Compatible strain evolution in two phases
565 due to epsilon martensite transformation in duplex TRIP-assisted stainless steels with
566 high hydrogen embrittlement resistance, *Int. J. Plast.* 88 (2017) 53–69.
- 567 [22] B.L. Ennis, E. Jimenez-Melero, E.H. Atzema, M. Krugla, M.A. Azeem, D. Rowley, D.

568 Daisenberger, D.N. Hanlon, P.D. Lee, Metastable austenite driven work-hardening
569 behaviour in a TRIP-assisted dual phase steel, *Int. J. Plast.* 88 (2017) 126–139.

570 [23] J.Y. Choi, J.H. Ji, S.W. Hwang, K.-T. Park, Strain induced martensitic transformation
571 of Fe–20Cr–5Mn–0.2Ni duplex stainless steel during cold rolling: Effects of nitrogen
572 addition, *Mater. Sci. Eng. A.* 528 (2011) 6012–6019.

573 [24] P.J. Jacques, Q. Furnémont, F. Lani, T. Pardoen, F. Delannay, Multiscale mechanics of
574 TRIP-assisted multiphase steels: I. Characterization and mechanical testing, *Acta*
575 *Mater.* 55 (2007) 3681–3693.

576 [25] Y. Tian, O.I. Gorbato, A. Borgenstam, A. V. Ruban, P. Hedström, Deformation
577 Microstructure and Deformation-Induced Martensite in Austenitic Fe–Cr–Ni Alloys
578 Depending on Stacking Fault Energy, *Metall. Mater. Trans. A.* 48 (2017) 1–7.

579 [26] O. Muránsky, P. Šittner, J. Zrník, E.C. Oliver, In situ neutron diffraction investigation
580 of the collaborative deformation–transformation mechanism in TRIP-assisted steels at
581 room and elevated temperatures, *Acta Mater.* 56 (2008) 3367–3379.

582 [27] J.Y. Choi, J.H. Ji, S.W. Hwang, K.-T. Park, Effects of nitrogen content on TRIP of
583 Fe–20Cr–5Mn–xN duplex stainless steel, *Mater. Sci. Eng. A.* 534 (2012) 673–680.

584 [28] P.A. Shade, B. Blank, J.C. Schuren, T.J. Turner, P. Kenesei, K. Goetze, R.M. Suter, J.
585 V. Bernier, S.F. Li, J. Lind, U. Lienert, J. Almer, A rotational and axial motion system
586 load frame insert for in situ high energy x-ray studies, *Rev. Sci. Instrum.* 86 (2015).

587 [29] B.H. Toby, R.B. Von Dreele, GSAS-II: The genesis of a modern open-source all
588 purpose crystallography software package, *J. Appl. Crystallogr.* 46 (2013) 544–549.

589 [30] A.K. De, D.C. Murdock, M.C. Mataya, J.G. Speer, D.K. Matlock, Quantitative
590 measurement of deformation-induced martensite in 304 stainless steel by X-ray
591 diffraction, *Scr. Mater.* 50 (2004) 1445–1449.

592 [31] M.J. Dickson, The Significance of Texture Parameters in Phase Analysis By X-Ray
593 Diffraction, *J. Appl. Crystallogr.* 2 (1969) 176–180.

594 [32] N. Jia, Z.H. Cong, X. Sun, S. Cheng, Z.H. Nie, Y. Ren, P.K. Liaw, Y.D. Wang, An in
595 situ high-energy X-ray diffraction study of micromechanical behavior of multiple
596 phases in advanced high-strength steels, *Acta Mater.* 57 (2009) 3965–3977.

597 [33] H.-R. Wenk, L. Lutterotti, P. Kaercher, W. Kaniupanyacharoen, L. Miyagi, R. Vasin,
598 Rietveld texture analysis from synchrotron diffraction images. II. Complex multiphase
599 materials and diamond anvil cell experiments, *Powder Diffr.* 29 (2014) 220–232.

600 [34] L. Lutterotti, R. Vasin, H.-R. Wenk, Rietveld texture analysis from synchrotron
601 diffraction images. I. Calibration and basic analysis, *Powder Diffr.* 29 (2014) 76–84.

602 [35] S. Matthies, H.G. Priesmeyer, M.R. Daymond, On the diffractive determination of
603 single-crystal elastic constants using polycrystalline samples, *J. Appl. Crystallogr.* 34
604 (2001) 585–601.

605 [36] B. Clausen, T. Lorentzen, T. Leffers, Self-consistent modeling of the plastic
606 deformation of f.c.c polycrystals and its implications for diffraction measurements of
607 internal stresses, *Acta Mater.* 9 (1998) 3087–3098.

608 [37] B.E. Warren, X-ray diffraction, Dover Publications Inc., New York, 1990.

609 [38] N.C. Popa, The (hkl) Dependence of Diffraction-Line Broadening Caused by Strain
610 and Size for all Laue Groups in Rietveld Refinement, *J. Appl. Crystallogr.* 31 (1998)
611 176–180.

612 [39] Y. Ma, W. Song, W. Bleck, Investigation of the Microstructure Evolution in a Fe-
613 17Mn-1.5Al-0.3C Steel via In Situ Synchrotron X-ray Diffraction during a Tensile
614 Test, *Materials* (Basel). 10 (2017) 1129.

615 [40] K. Inal, J.L. Lebrun, M. Belassel, Second-order stresses and strains in heterogeneous
616 steels: Self-consistent modeling and X-ray diffraction analysis, *Metall. Mater. Trans.*
617 *A.* 35 (2004) 2361–2369.

618 [41] S. Zaefferer, N.-N. Elhami, Theory and application of electron channelling contrast
619 imaging under controlled diffraction conditions, *Acta Mater.* 75 (2014) 20–50.

620 [42] F. Bachmann, R. Hielscher, H. Schaeben, Texture Analysis with MTEX – Free and
621 Open Source Software Toolbox, *Solid State Phenom.* 160 (2010) 63–68.

622 [43] J. Goulden, K. Mehnert, K. Thomsen, H. Jiang, A Method for Separating
623 Crystallographically Similar Phases in Steels using EBSD and Machine Learning,
624 *Microsc. Microanal.* 23 (2017) 110–111.

625 [44] G.B. Olson, M. Cohen, A general mechanism of martensitic nucleation: Part I. General
626 concepts and the FCC → HCP transformation, *Metall. Trans. A.* 7 (1976) 1897–1904.

627 [45] P. Ferreira, P. Müllner, A thermodynamic model for the stacking-fault energy, *Acta*
628 *Mater.* 46 (1998) 4479–4484.

629 [46] S. Allain, J.P. Chateau, O. Bouaziz, S. Migot, N. Guelton, Correlations between the
630 calculated stacking fault energy and the plasticity mechanisms in Fe-Mn-C alloys,
631 *Mater. Sci. Eng. A.* 387–389 (2004) 158–162.

632 [47] J. Andersson, T. Helander, L. Höglund, Thermo-Calc & DICTRA, computational tools
633 for materials science, *Calphad.* 26 (2002) 273–312.

634 [48] TCSAB: Tcfe8-tcs steels/fe-alloys database 8., n.d.

635 [49] E.C. Oliver, M.R. Daymond, P.J. Withers, Interphase and intergranular stress
636 generation in carbon steels, *Acta Mater.* 52 (2004) 1937–1951.

637 [50] R.R. Shen, On the low primary water stress corrosion cracking susceptibility of weld
638 deformed Alloy 690, KTH Royal Institute of Technology, 2017.

639 [51] J.Y. Choi, J. Lee, K. Lee, J.-Y. Koh, J.-H. Cho, H.N. Han, K.-T. Park, Effects of the
640 strain rate on the tensile properties of a TRIP-aided duplex stainless steel, *Mater. Sci.*
641 *Eng. A.* 666 (2016) 280–287.

642 [52] Y. Tian, U. Lienert, A. Borgenstam, T. Fischer, P. Hedström, Martensite formation
643 during incremental cooling of Fe-Cr-Ni alloys: An in-situ bulk X-ray study of the
644 grain-averaged and single-grain behavior, *Scr. Mater.* 136 (2017) 124–127.

645 [53] G. Olson, M. Cohen, A mechanism for the strain-induced nucleation of martensitic
646 transformations, *J. Less Common Met.* 28 (1972) 107–118.

647 [54] A.J. Bogers, W.G. Burgers, Partial dislocations on the {110} planes in the B.C.C.
648 lattice and the transition of the F.C.C. into the B.C.C. lattice, *Acta Metall.* 12 (1964)
649 255–261.

650 [55] N. Nakada, H. Ito, Y. Matsuoka, T. Tsuchiyama, S. Takaki, Deformation-induced
651 martensitic transformation behavior in cold-rolled and cold-drawn type 316 stainless
652 steels, *Acta Mater.* 58 (2010) 895–903.

653 [56] T. Suzuki, H. Kojima, K. Suzuki, T. Hashimoto, S. Koike, M. Ichihara, Plastic
654 deformation and martensitic transformation in an iron-base alloy, *Scr. Metall.* 10
655 (1976) 353–358.

- 656 [57] J. Talonen, Effect of Strain-Induced α' -Martensite Transformation on Mechanical
657 Properties of Metastable Austenitic Stainless Steels, Helsinki University of
658 Technology, 2007.
- 659 [58] P. Hedström, L.E. Lindgren, J. Almer, U. Lienert, J. Bernier, M. Turner, M. Odén,
660 Load Partitioning and Strain-Induced Martensite Formation during Tensile Loading of
661 a Metastable Austenitic Stainless Steel, Metall. Mater. Trans. A. 40 (2009) 1039–
662 1048.
- 663