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Zillmann, B.:

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Experimental characterization of microstructure development during loading path changes in bcc sheet steels

T. Clausmeyer · G. Gerstein · S. Bargmann · B. Svendsen · A.H. van den Boogaard · B. Zillmann

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Abstract Interstitial free sheet steels show transient work hardening behavior, i.e. the Bauschinger effect and cross hardening after changes in loading path. This behavior affects sheet forming processes and the properties of the final part. The transient work hardening behavior is attributed to changes in the dislocation structure. In this work, the morphology of the dislocation microstructure is investigated for uniaxial and plane strain tension, monotonic and forward to reverse shear, as well as plane strain tension to shear. Characteristic features such as the thickness of cell walls and the shape of cells are used to distinguish microstructural patterns corresponding to different loading paths. The influence of crystallographic texture on the dislocation structure

is analysed. Digital image processing is used to create a “library” of schematic representations of the dislocation microstructure. The dislocation microstructures corresponding to uniaxial tension, plane strain tension, monotonic shear, forward to reverse shear and plane strain tension to shear can be distinguished from each other based on the thickness of cell walls and the shape of cells. A statistical analysis of the wall thickness distribution shows that the wall thickness decreases with increasing deformation and that there are differences between simple shear and uniaxial tension. A change in loading path leads to changes in the dislocation structure. The knowledge of the specific features of the dislocation structure corresponding to a loading path may be used for two purposes: (i) the analysis of the homogeneity of deformation in a test sample and (ii) the analysis of a formed part.

Keywords Electron microscopy · Steel · Dislocations · Mechanical testing · Sheet forming

T. Clausmeyer
Institute of Mechanics, TU Dortmund University, Leonhard-Euler-Str. 5, 44227 Dortmund, Germany
Tel.: +49-231-755-5633
Fax: +49-231-755-5633
E-mail: till.clausmeyer@udo.edu

G. Gerstein
Institute of Material Science, Leibniz Universität Hannover, An der Universität 2, 30823 Hannover, Germany

S. Bargmann
Institute of Continuum and Material Mechanics, TU Hamburg-Harburg University, Eißendorfer Str. 42, 21703 Hamburg, Germany, Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Max-Planck-Str. 1, 21502 Geesthacht, Germany

B. Svendsen
Material Mechanics, Jülich Aachen Research Alliance, RWTH Aachen University, Schinkelstr. 2, 52062 Aachen, Germany

A.H. van den Boogaard
Faculty of Engineering Technology, University of Twente, P.O. Box 217, 7500 AE Enschede, The Netherlands

B. Zillmann
Institute of Materials Science and Engineering, TU Chemnitz University, Erfenschlager Str. 73, 09125 Chemnitz, Germany

1 Introduction

Metal forming processes generally involve large deformation and one or more loading-path changes. In many cases, this may result in the development of oriented dislocation structures as e.g. shown in [1, 2] which result in anisotropic flow, hardening, and complex stress-deformation behavior. Among the first to investigate the effect of different loading paths on microstructure evolution, Fernandes and Schmitt [3] examined the microstructure and glide system activity resulting from uniaxial tension, plane-strain tension, biaxial tension, as well as uniaxial to biaxial tension loading-path changes in low carbon steel. Their results demonstrated that newly created dislocation structures in the second stage of two-stage deformation tests are similar to the dislocation

structures observed in monotonic tests. In addition, a saturation of the dislocation microstructure was observed for large strain amplitudes after path changes. Here and in the following, the term saturation of microstructure means that the morphology, especially the shape, of dislocations patterns does not evolve with increasing monotonic deformation once a particular pattern has been established. Pantleon and Stoyan [4] supported this concept with a statistical model for the correlation or more precisely anti-correlation of the misorientation angles across a large number of dislocation boundaries. Their results can be interpreted in the sense that there might be an ordering mechanisms governing the formation of dislocation boundaries. Rauch and Schmitt [5] established links between the macroscopic tests involving loading path changes and the observed microstructure. In particular, they investigated low carbon steel subjected to different sequences of simple shear. Lins et al. [6] characterized the microstructure of interstitial free steel after hot rolling with SEM and TEM methods. Wilson and Bate [7] conclude in their study on low carbon steel that the effect of grain morphology and texture on phenomena like cross hardening is small compared to the effect of the dislocation structure. Nesterova and co-workers [1, 2] carried out an x-ray diffraction investigation of dislocation structures developing in low carbon steel during two-stage tests involving different sequences of simple shear and uniaxial tension to simple shear. An evolution of certain components of the γ -fiber was observed for large shear and reverse shear in their studies. Nesterova et al. observed differences in the morphology of dislocation walls in their shear tests depending on the particular orientation within the γ -fiber of a grain. Vincze et al. [8] conducted tests with strain reversal in combination with variations in the ambient temperature (20° C and -120° C) for the pre-straining and loading reversal steps. Tests at low temperature were carried out to impede dislocation movement and corresponding patterning. Vincze et al. “concluded that the physical process responsible for the transient stagnation of strain hardening is related to the nature of the dislocations generated during the prestrain and [to] their evolutionary law during reloading”, such that “the mechanical behaviour is controlled by individual interactions between the moving dislocations and the obstacles lying on their slip planes rather than [by] some cell structure evolution”.

In all investigations of low-carbon steel specimens subjected to load reversal, dissolution of dislocation cell structures upon load reversal was observed. Such an effect was also observed earlier by Hasegawa et al. [9] in commercially pure aluminum. In particular, several authors concluded that dissolution of pre-existing cell structures is responsible for the observed hardening stagnation and softening after load reversal [1, 2, 5, 7, 10, 14]. Figueiredo et al. [11] also showed that cyclic bending influences the mechanical properties of

drawn bars manufactured from AISI 1010 steel. The correlation of the strain path and the dislocation structures resulting therefrom was also investigated for fcc material by Ding et al. [12] and Landau et al. [13] for an aluminum alloy and copper, respectively. Based on tests at different temperatures, Vincze et al. [8] additionally concluded that load reversal also affects the dislocation annihilation rate and / or the mean-free path. Cross hardening was attributed to the fact that dislocation structures which formed during the first loading stage represent additional obstacles for the activation of new glide systems at the start of the second loading stage. Loading-driven glide of these new systems then results in a breakdown and dissolution of the “old” dislocation structures formed during the previous loading stage, representing hardening stagnation and softening in stress-strain space. Dislocation glide in the new loading direction results in the build-up of dislocation structures aligned with respect to the new loading direction, analogously to the case of monotonic loading in the new loading direction from the start. The understanding of these microstructural interactions is essential to the development of phenomenological polycrystal e.g. [15–18] and crystal plasticity models e.g. [19, 20] for anisotropic and, in particular, cross hardening. In this context, Beyerlein et al. [21] analysed transient hardening in copper during equal channel angular extrusion with the help of a crystal plasticity model. Wang et al. [22] showed that cross hardening may have an effect on the geometry of split rings after springback. The investigated rings were cut out of an axis-symmetric cup manufactured by sheet metal forming. Thuillier et al. [23] observed an influence of the Bauschinger effect and hardening stagnation on the punch force during a two-stage drawing process.

Based on macroscopic testing and microstructure characterization by x-ray-diffraction (XRD) and TEM, the purpose of the current work is the investigation of the stress-deformation behavior and microstructure development in an interstitial-free bcc steel as a function of the type of loading path and crystallographic orientation. In a comprehensive analysis the morphologies of dislocation structures reported in different previous works are compared with the current investigations for additional loadings. This serves the purpose of establishing a comprehensive overview of morphological features corresponding to a particular loading path. The loading paths investigated in the current work include (i) monotonic loading in simple shear, (ii) forward-reverse (cyclic) loading in simple shear, and (iii) orthogonal loading from plane-strain tension to simple shear. In particular, the latter is in contrast to previous studies, which focused on uniaxial rather than plane-strain tension. Although the stress-deformation behavior of DC06 for this latter type of loading has already been investigated [24], the corresponding microstructure development in DC06 has not been reported on in the literature. The present work contributes to

the creation of a “library” of representative dislocation microstructures and their corresponding stress-deformation relation for the class of ferritic interstitial free steels. Attention is paid to the analysis of the morphology of larger scale dislocation structures as dense dislocation walls, as well as to the morphology of smaller scale single dislocation cells. Strain levels relevant for sheet metal forming operations, i.e. equivalent strains of 0 to ≈ 0.3 are investigated. Sheet metal parts having experienced such characteristic levels of deformation are investigated in the literature e.g. in [25]. For this purpose, schematics of the dislocation microstructure are created with the help of image processing in order to highlight the important features for different loadings. The wall thickness distribution for uniaxial tension and simple shear and its evolution with increasing strain is analysed statistically.

At first, basic properties of the tested material DC06 and the result of the mechanical tests are discussed. The terminology for the description of the dislocation microstructure is introduced. The initial texture and the evolution of texture is analysed for deformation modes relevant for this work. Characteristic and representative features of the dislocation microstructure are described on the basis of actual TEM-micrographs and their corresponding schematics. Here, representative means that the investigated crystals have orientations which are representative of the material. In this context, the crystallographic texture is interpreted as a statistical quantity inherent to the material. The investigated characteristic features are the thickness of cell walls, the number of cells per grain and the shape of the cells. The correlation between the dislocation microstructure and the observed transient behavior in stress-deformation relations is analyzed. The work concludes with a discussion of the observed findings and their relevance.

2 Material testing

2.1 Material

The interstitial free steel DC06 belongs to the category of cold rolled low carbon steel processed to flat products and is used for cold forming. It is a sheet steel quality mostly manufactured for the automobile industry by ThyssenKrupp Steel Europe. Table 1 states the chemical composition of the interstitial free steel DC06 (thickness 1.00 mm) delivered by ThyssenKrupp Steel Europe AG according to [26].

C	Si	Mn	P	S	Al	N	Ti
3	18	137	13	10	35	2.7	79

Table 1 Chemical composition of DC06 according to [26]. All values are given as multiple of 10^{-5} mass fraction and were determined by ThyssenKrupp Steel Europe [27].

After cold-rolling the material was annealed and subjected to a final skin-pass by the manufacturer. The average Young's Modulus E over the three directions 0° , 45° and 90° with respect to the rolling direction is determined as 181 000 MPa with Poisson's ratio $\nu = 0.3$. The initial texture is a fibre texture with the $\langle 111 \rangle$ direction oriented parallel to the sheet normal direction. The average grain size is $20 \mu\text{m}$ with single grain sizes ranging from 5 to $60 \mu\text{m}$.

Average r -values are computed by evaluation of the ratios of total plastic strain for the beginning of plastic deformation (i.e., a plastic strain of 0.002) to the uniform strain (i.e., the strain before necking), resulting in $r_0 = 2.31$, $r_{45} = 1.95$, $r_{90} = 2.77$. All test specimens used in this work were prepared from the same batch.

2.2 Types of testing

In the current work, testing results were obtained for the following loading programs: (i) uniaxial tension at 0° , 45° and 90° to the rolling direction, (ii) plane-strain tension, (iii) monotonic and cyclic simple shear, (iv) plane-strain tension to shear orthogonal loading. The uniaxial tension tests were performed at the Institute of Material Science and Engineering in Chemnitz. The plane-strain tension tests were performed on the biaxial tester at the Faculty of Engineering Technology (CTW), University of Twente. In particular, this latter device is capable of loading a sheet metal specimen in both simple shear and in plane-strain tension.

It consists of a regular uniaxial testing device which is used to achieve plane-strain tension in the sample via an actuator. A subframe mounted between the cross bars accommodates the actuator for simple shear deformation. The deformation is applied to the sample as indicated in Fig. 1.

Fig. 1 Biaxial test setup. Geometry of the tension-shear specimen and the measurement region of height 3.0 mm and width 45.0 mm. The checked region indicates the actual specimen and the black area marks the actual deformation zone. The tension direction is direction 2 and the shear direction is direction 1.

The ratio of the height of the deformation region to the sample thickness is chosen in order to minimize the chance of buckling during simple shear, here 3:1. Furthermore, in order to achieve homogeneous deformation in the measurement area, the ratio of the width to the height is large, i.e. in this case 15:1, as shown in Fig. 1. The deformation is measured in an area within the deformation zone (see the magnified region on the right of Fig. 1).

The deformation field $x_i = \chi_i(X_J)$ in the material during the tests is determined by an optical measurement system described in [24, 28]. Here, X_J and x_i denote the coordinates of material points in the initial (undeformed) and current (deformed) configuration, respectively. From this, the components $F_{iJ}(X_J) = \partial \chi_i(X_J) / \partial X_J$ of the deformation gradient are determined. Analogously, the force measurements are represented in terms of the components T_{ij} of the Cauchy stress, i.e., force per unit current area. The current area is computed by exploiting the relation $\det(\mathbf{F}) = 1$ for isochoric deformation. Further details on the experimental setup can be found in [24].

2.3 Testing results

Consider first the results for uniaxial and plane-strain tension shown in Fig. 2. The yield stress in plane strain tension is about 18 % higher than in the uniaxial tension case. Also, due to the different kinematics and the anisotropy of the material, the hardening behavior observed in Fig. 2 is different. This is consistent with earlier results for IF-steel obtained by [29]. In the current work, attention is focused on the investigation of the loading-path dependence of the material behavior and the concomitant microstructure development under quasi-static conditions.

Consider next the case of load reversal in simple shear as shown in Fig. 3.

Fig. 2 Cauchy normal stress T_{22} as a function of $F_{22} - 1$ for uniaxial tension (uni ten) and plane-strain tension (ps ten) at 10^{-3} s^{-1} (quasi-static).

Fig. 3 Cauchy shear stress T_{12} as a function of F_{12} for load reversal after shear up to 13% (cyc 1), 49% (cyc 2), and 67% (cyc 3). See text for discussion.

In accordance with earlier observations e.g. [24, 30, 31] for interstitial free steels like DC06, the material exhibits the Bauschinger effect [32], i.e., early re-yielding after unloading and load reversal, followed by hardening stagnation. The levels of kinematic hardening and hardening stagnation are clearly dependent on the amount of pre-shear. A difference of approximately 50 MPa for the largest amount of pre-shear can be observed ($T_{12} = 225 \text{ MPa}$ before load reversal and $T_{12} \approx 175 \text{ MPa}$ at re-yielding). Finally, results for plane-strain tension to simple shear orthogonal loading are shown in Fig. 4.

Fig. 4 Shear stress T_{12} as a function of $F_{22} + F_{12} - 1$ (plane-strain tension to simple shear).

The plane strain tension to simple shear deformation was obtained via unloading after the tension phase (10 %) and after reloading into the shear phase. To gain insight into the reason for these tendencies in the stress and deformation behavior as a function of loading path, we now turn to the investigation of the corresponding microstructure development. In this context it is important to mention that the transient work hardening behavior after load reversal or an orthogonal loading path change in interstitial free steels affects the forming process and the properties of the final parts [22, 23]. The current deals with the question as to which extent the knowledge of the specific features of the dislocation microstructure can be used in the context of sheet metal forming.

3 Microstructure investigation

3.1 TEM sample preparation

The dislocation microstructure development due to loading is investigated in this work with the help of transmission electron microscopy (TEM). To this end, TEM foils in the form of flat discs with a diameter of 3 mm and a thickness of approximately 500 μm were cut from the center of the deformation zone of the deformed sample (Fig. 1) by wire eroding. These disks were mechanically thinned down to 100 μm and electropolished on both sides with an electrolyte consisting of 120 ml 40 % perchloric acid, 440 ml butoxyethanol and 440 ml 100 % acetic acid in a Struers Tenu Pol 5 electrolytic polishing machine. All TEM investigations were conducted on a JEOL JEM2010 transmission electron microscope with a 200 kV electron gun.

3.2 Texture analysis

In addition to the characterization of the local orientation with the help of the diffractions patterns gained by TEM analysis, the bulk texture of the material was characterized using x-ray diffraction (XRD) at the Institute of Materials Science and Engineering, TU Chemnitz University. For this a Siemens D5000 diffractometer operating at 40 kV and 15 mA (Co-K α radiation) was used to measure three incomplete pole figures of the $\{200\}$, $\{220\}$ and $\{211\}$ planes.

The samples were cut from the center of the specimen along the sheet thickness. Orientation distribution functions (ODF) were computed from the raw data with the MTEX [33] software. The MTEX algorithm is based upon the minimization of a least-square functional. Between 9000 and 13000 orientations were processed per sample. The Bunge convention for the Euler angles is used in this work. Sample symmetries are not exploited such that, in the Euler angle representation of the ODF, φ_1 ranges from 0° to 360° .

The analysis of the texture in the as-received state and in deformed states (uniaxial tension, plane strain tension, simple shear) was performed to identify the crystallographic orientations which are relevant for the bulk material in a statistical sense. It is analyzed how the deformations mentioned affect the initial texture. For this purpose the φ_2 sections of the Euler space are analyzed.

The nomenclature used in [1, 2] is mainly adopted in the description of the textures and the corresponding analysis of the evolution of the dislocation microstructure. In this context, the first and second reference axes of the sample system are the tension direction (TenD) and the transverse direction (TD) for the investigated tension cases. The tension direction (TenD) was aligned with the rolling direction (RD) for the samples discussed in this work. The first and second reference axes of the sample system are the shear direction (SD) and the shear plane normal (SPN) for the texture analysis shown in Fig. 5. In this case the rolling direction (RD) coincides with the shear (direction). As mentioned before, in the as-received state there is a strong γ -fiber (i.e. the $\langle 111 \rangle$ direction is parallel to ND or $\varphi_2 = 45^\circ$, $\phi \approx 55^\circ \pm 5^\circ$) which is characteristic of cold-rolled IF steel.

Fig. 5 Euler angle representation of ODF computed from the measured diffraction data along the $\phi_2=45^\circ$ sections. All angles are given in degree.

Plane strain tension in the rolling direction leads to an increase of the intensity of the rolling texture with a strengthening of the γ_2 -component ($\phi_1 = 0^\circ$, consistent with the terminology used in section III of [2]) as Fig. 5(c) shows. The same tendency with a stronger concentration around $\phi_2 = 0^\circ$ and $\phi_2 = 60^\circ$ is observed for uniaxial tension (see Fig. 5(b)). Simple shear along the rolling direction leads to a reorientation mainly of the γ_2 -component and a strengthening of the γ_1 - component ($\phi_1 = 30^\circ$) as reported in [2]. Nesterova et al. [2] performed texture simulations with a simplified full-constraints Taylor model to analyse the re-orientation path of orientations belonging to the γ -fiber. On the basis of the texture analysis of the material investigated in this work, we conclude that it shows qualitatively comparable texture evolution to the IF-steel investigated in [2]. Earlier results of Bacroix and Hu [34] show that the final texture after shear is independent of the direction of shear in the plane described by the rolling direction and the transverse direction. Since

the γ -fiber and its components are representative of the material, the TEM analysis focuses on γ -grains.

3.3 Characterization of dislocation microstructure

For the description of the dislocation microstructure, the following terminology is used which is mainly adopted from [35, 36]. Two types of dislocation boundaries are distinguished based on their formation: *geometrically necessary boundaries* (GNBs) and *geometrically incidental boundaries* (IBs). The GNBs are necessary to accommodate misorientations induced by differences in dislocation glide in different regions of a volume element according to [36]. Here, the *misorientation* between two elements of the microstructure refers to the change in local lattice orientation. The IBs are boundaries that form due to “statistical mutual trapping of glide dislocations often supplemented by ‘forest dislocations’ ” according to [37]. In the specimens analysed for this work the misorientation between an IB and adjacent regions is close to the range of the maximum accuracy of the measurement method. In this work misorientations were determined by interpretation of diffraction patterns in TEM images. For the current investigations the maximum accuracy for the determination of misorientations lies between 0.5° and 0.8° . Both types of boundaries have a considerably larger dislocation density than the adjacent regions. Based on their appearance in TEM micrographs, the following differentiations are made: A (dislocation) *cell interior* (CI) is defined as a region of lowest dislocation density and basically uniform dislocation distribution. These cell interiors are surrounded and separated from other cells by regions of higher dislocation density termed (dislocation) *cell walls* (CWs). The change in local lattice orientation from a cell to a wall, referred to in this work as the *misorientation* between these, falls in the range of 1.0° to 1.5° . This means that cell walls (CWs) are an example for incidental boundaries (IBs). Cell walls may confine CIs. Walls with the highest dislocation density are called *dense dislocation walls* (DDWs). They belong to the group of GNBs. In the investigated samples the misorientation between DDWs and adjacent regions is at least 1° . Since the accuracy of the method used for determining the misorientation falls in this range, a determination of an absolute value is problematic.

A similar procedure as described by Rybin (pp. 23-24 of [44]) for the determination of misorientations was used. When we refer to the misorientation between a DDW and an adjacent region, it means that we measured the orientation within the DDW close to its edge and outside the DDW also very close to the edge. In a similar work Hughes and Hansen [51] determined a minimum average misorientation angle of 1.2° for an equivalent strain of 0.12 in nickel. The interpretation of the diffraction patterns analyzed in this work showed that the misorientations between GNBs and adjacent regions

are roughly three times larger than those of IBs and adjacent regions. In general, DDWs extend across several cells and can extend across grains. In turn, groups of cells and surrounding walls form so-called *cell blocks* (CBs) separated from each other by *cell-block boundaries* (CBBs). CBBs are a special case of DDWs which delimit cell blocks. With progressing deformation, double walls (DWs) form when a single DDW splits into (two) separate walls.

3.4 TEM analysis procedure

As mentioned before, the analysed TEM foils have a diameter of 3 mm with the diameter of the hole approx. being 1 mm. The zone which can be effectively investigated extends radially 30 μm into the disk, so that approx. 100 000 μm^2 of each foil can be investigated. Of this total area approx. 1/3 can be analysed in detail because the illumination conditions in the regions close to the hole and further away from the hole are not sufficient. Before proceeding with the analysis the diffraction pattern was checked for symmetry. With an average grain size of 20 μm an average grain area of approx. 300 μm^2 is obtained.

This means that approx. 1000 grains can be investigated per foil. 50-100 images of such suitable and well-illuminated grains were taken. At least 80 % of the taken images show grains belonging to the γ -fiber. In the description of the crystallographic orientations of the TEM-images a simplified differentiation of the γ -fiber grains (i.e. a $\langle 111 \rangle$ direction is parallel to the normal direction) is made. In this work and a previous work [27] only grains belonging to the γ -fiber are shown.

In the plane strain and uniaxial tension tests a grain belongs to the γ_1 fiber if a $\langle 112 \rangle$ direction is within 10° to RD. In tension a grain belongs to the γ_2 fiber if a $\langle 110 \rangle$ is within 10° to RD. All other grains of the γ -fiber are referred to as general γ grains. Note, that in tension the γ_2 fiber is stable as the texture analysis (Fig. 5) has shown. This is consistent with the definition used in [2]. In shear, the two cases, i.e. where the shear direction (SD) is parallel to the RD or the SD is parallel to the transverse direction (TD), are distinguished as follows: If the rolling direction (RD) and the shear direction (SD) coincide, the aforementioned definitions for tension are adopted. If the SD coincides with the transverse direction (TD), we define the γ_1^* grains as those with a $\langle 112 \rangle$ direction within 10° to SD. Consequently, the γ_2^* grains are those with a $\langle 110 \rangle$ within 10° to SD. The definition of the general γ case is in analogy to the aforementioned cases.

For comparison purposes the equivalent strain α_P according to von Mises was computed and isochoric, ideal, isotropic plasticity was assumed. Table 2 lists the specimens which were analysed by TEM. Specimens of TEM images

shown in this work are marked with an asterisk *. The investigated levels of deformation are in the range relevant for sheet metal forming.

(a) tension in rolling direction						
	uniaxial				plane strain	
$F_{22} - 1$	0.05*	0.1	0.2*	0.3	0.08*	0.2*
α_P [%]	4.9	9.5	18.2	26.2	8.9	21.1

(b) monotonic simple shear					
	in RD		in TD		
F_{12}	0.1	0.3*	0.115*	0.3	0.5
α_P [%]	5.8	17.3	6.6	17.3	28.9

(c) loading with strain path change			
	forward $\rightarrow$ reverse shear	ps tension $\rightarrow$ shear	
$F_{22} - 1$	-	-	0.11*
F_{12}	0.12 $\rightarrow$ 0*	0.12 $\rightarrow$ 0.06	0.35*
α_P [%]	13.9	10.3	30.3

Table 2 Specimen analysed in TEM

The schematics shown in Figs. 7-14 were created by applying the digital image processing described above to the original images. In a previous work on cell structures by Juda et al. [38], the cell structure in GaAs and CaF₂ single crystals was investigated.

Fig. 6 shows the effect of the applied image processing for a single cell after 11.5 % shear ($\alpha_P = 6.6\%$) in the rolling direction. At first the contrast is adjusted so that 3 % of the pixels saturate at both ends of the intensity range, i.e. 0-255. The image is then binarized. The morphological operations opening and closing with a structuring element of a 3 by 3 cross were performed. The application of these operations in the context of microscopy is discussed in [39] and generally in [41]. As the last step of the automated process the function "Outline", an edge detection algorithm, of the program Image J [40] was used. As a final step manual corrections were made for the images shown in this work. A careful inspection of Fig. 6(b) and e.g. Fig. 7(b) shows that lines in the final schematics are straighter or more continuous leading to a less busy appearance. The line width of some lines (e.g. grain boundaries) was also modified in this regard because the line widths after the automated operation are rather thin as Fig. 6(b) shows. Such semi-automated processing of approx. 10 TEM images per deformation mode and increment in deformation was used in order to obtain information on the statistical relevance of observed features. In addition, an analysis of the statistical distribution of the thickness of DDWs was performed (see section 3.8).

Fig. 6 Effect of image processing for the example of a single cell after 11.5 % ($\alpha_p = 6.6\%$) shear in the rolling direction.

In the current work the dislocation density was determined as the projected, summed length of a given area per volume according to [42, 43]. This method was used for the determination of the dislocation density in CBBs when single dislocation lines could be distinguished at a suitable magnification. In the case of very distinct CBBs an estimation of the dislocation density in CBBs was performed. The basis for this was the relative gray level in the TEM image compared to other regions in the same image.

3.5 Plane-strain & uniaxial tension

During uniaxial tension of DC06 at room temperature, CWs begin to form from dislocation tangles at about 5% deformation. After approx. 12% tension, basically all tangles are incorporated into CWs or DDWs. Further deformation results in an increase in the misorientation angle between CIs and CWs or CBBs. The formation of such CBBs might induce the formation of sharp boundaries referred to as blade-like plates [44] on the level of several grains (10-100 μm). Solid white lines with plain arrows indicate crystallographic directions and white lines indicate traces of crystallographic planes in the micrographs. Fig. 7 shows a region at 5% deformation (equivalent strain $\alpha_p=4.9\%$) in uniaxial tension, and Fig. 9 shows a region at 8% deformation ($\alpha_p=8.9\%$) in plane-strain tension. Both these grains belong to γ_2 -fiber which is characteristic for tension deformation. Note, that the CBBs are much more pronounced at 20 % tension (Fig. 8).

Fig. 7 Fragmentation of CI-CW dislocation structure into CBs and incipient CBBs (emphasized by the dashed lines) at 5% ($\alpha_p = 4.9\%$) deformation in DC06 subject to uniaxial tension in the RD, which is the same as the tension direction (TenD). Single CBB segments are rather straight in this γ_2 grain.. A single cell wall which does not encompass a closed segment is marked as CW. The circle (in (b)) highlights a cell interior interspersed with incidental dislocation boundaries. The dashed lines in (b) highlight preferred directions of CBBs.

The CBBs in plane strain tension at higher deformation (see Fig. 10) shows stronger curvature than in the uniaxial case. In particular, the alignment of the CBBs with the tension direction (TenD) is more pronounced in the uniaxial (Fig. 7) than in the biaxial (Fig. 9) case. This is just one indication of the different stress states acting in the two cases.

Fig. 8 Characteristic CBBs after 20 % ($\alpha_p = 18.2\%$) uniaxial tension in the RD for the central γ_2 -grain.

During tension, CBBs initially form with respect to $\{110\}$ systems. As loading proceeds, CBBs associated with $\{112\}$, and above 15% deformation, with $\{123\}$, systems, can also be observed. Activity on $\{110\}$ systems was also reported in [46, 47], while [48] reported activity on $\{123\}$ in iron. As suggested in particular by the uniaxial results in Fig. 7, the formation of the incipient CB-CBB dislocation structure may be referred to as fragmentation. The intensity of fragmentation, i.e. the number of CBs per volume or area, is relatively high in the uniaxial case and not as high for plane-strain.

The CBBs in the uniaxial case are sharper, meaning that they are not as thick (average of ≈ 100 nm in Fig. 7. compared to an average of ≈ 300 nm in Fig. 9). A more detailed analysis of the distribution of DDWs is presented in the next section. In addition, the curvature of single CBB segments is higher in plain strain tension (see e.g. the horizontal CBBs in the center of Fig. 9). The dislocation density in the CBBs in plane strain tension is lower than in the uniaxial case. In summary, the CBBs in plane strain tension seem to be more diffuse. The CBBs are usually aligned with the macroscopic loading direction, e.g., the tension (TenD) and rolling direction (RD) in Fig. 7.

For plane strain tension CBBs are less well-aligned with the loading-rolling direction (TenD=RD), than in the case of uniaxial tension. For the same normal strain the number of cells is twice as high in uniaxial tension compared to plane strain tension. Other plane strain and uniaxial tension samples investigated in this work confirm that the appearance of the dislocation structure in plane strain tension becomes more similar to the uniaxial tension case for higher applied strains.

In some grains CBBs become more diffuse, i.e. they show stronger curvature and single segments are shorter, when they are close to a grain boundary. This is especially the case when the orientation between the neighboring grains is large.

(a) Micrograph

(b) Schematic

Fig. 9 Fragmentation of CI-CW dislocation structure into CBs and CBBs at 8% deformation in DC06 subject to plane-strain tension in the RD, which corresponds here with the tension direction (TenD). The central grain belongs to the general γ_2 -fiber. The formation of CBBs is stronger in the vertical direction than in the horizontal direction. The segments of CBBs show a stronger curvature than in uniaxial tension.

Fig. 10 Characteristic CBBs for plane strain tension after 20 % ($\alpha_p = 21.1\%$) plane strain tension in the RD for γ_2 -grain.

3.6 Monotonic & reverse shear

3.6.1 Monotonic shear

In the case of simple monotonic shear, CBBs are thicker than in uniaxial tension (average thickness in Fig. 7 ≈ 100 nm compared to 250 nm in Fig. 12) and slightly thinner than in the plane-strain tension case (average thickness in Fig. 9 ≈ 300 nm). This is discussed in more detail in the next section. The micrograph shown in Fig. 12) was prepared from a sample subjected to monotonic shear of 12 % ($\alpha_p = 6.6\%$). The grain belongs to the general γ fiber.

(a) Micrograph

(b) Schematic

Fig. 12 Dislocation microstructure after 11.5 % ($\alpha_p = 6.6\%$) monotonic simple shear in the transverse direction. The right grain belongs to the general γ - fiber. The schematic emphasizes the characteristic rectangular, almost square shape of CBBs in the monotonic simple shear case. The CBBs tend to be thicker (average thickness ≈ 250 nm) than in the uniaxial tension case (average thickness ≈ 100 nm). The ellipse in (a) highlights a cell which containing a number of single dislocations.

Fig. 11 Two families of CBBs after 30 % ($\alpha_p = 17.3\%$) simple shear in the RD for a γ_1 -grain.

In addition, the CBs are more square with sides parallel and perpendicular to the loading direction. Single dislocations can be observed less frequently in CBs (e.g. the CB highlighted with a circle in Fig. 12(a)) than in the tension case. The number of cells per area is about 1.5 times as high as in plane strain tension at comparable levels of the deformation. In simple shear a stronger influence of the orientation

of single grains is observed. The deformed γ_1 -grain (30 % shear in the RD=SD) shown in Fig. 11 shows similar rectangular cells as the one in Fig. 12. However, the inclination of the DDWs with respect to the macroscopic axes are different. In the γ_1 in Fig. 11 one family of DDWs is parallel to the SD, while in the general γ -grain the shear direction is inclined approx. 45° to the SD. Note, that the direction of the shear direction with respect to the RD differs in these two grains. The occurrence of two families of DDWs in γ_1 -grains is not frequent. γ_1 -grains often show one family of DDWs. Fig. 6 in [27] shows a γ_2^* -grain of a specimen after 30 % shear in the TD from the same set of experiments as was considered in this work. The grain shows one distinct family of DDWs roughly inclined 45° to SD. The CBs within the CBBs are also rectangular, almost square. Similar to the tension case, two families of DDWs are observed more frequently in the vicinity of grain boundaries. As in the tension case this hints at an indirect influence of the grain size, i.e. with decreasing grain size these effects can be observed more frequently.

3.6.2 Reverse shear

The dislocation microstructure after moderate amounts of forward shear and moderate amounts of reverse shear shares similarities with the monotonic shear case. The micrograph shown in Fig. 13(b) was prepared from a sample subjected to 12 % forward and 12 % reverse shear (resulting $\alpha_P = 13.9\%$). The grain belongs to the γ_1 -fiber. Due to the relatively small amplitudes of applied shear and the reversal of deformation, it can be assumed that the grain also initially belonged to the γ -fiber. The underlying rectangular pattern of CBBs is also visible in the central region in the schematic of Fig. 13(b).

However, reversal of the direction of deformation leads to partial disintegration and distortion of the dislocation microstructure created in forward shear. In the lower left-hand corner of Fig. 13(a) it appears that a DDW was partly disintegrated (highlighted by the white ellipse) because the traces of a former DDW are clearly visible outside, but not inside the ellipse. A section of the mentioned DDW has split into two double walls (DWs) above the section where the DDW vanished. Another partly disintegrated DDW is highlighted by the second white ellipse in the center of Fig. 13(a). In the top center of Fig. 13(b) a black ellipse highlights a single cell block. It can be assumed that the corresponding CB had initially been rectangular, but was distorted after the reversal of the deformation.

(a) Micrograph

(b) Schematic

Fig. 13 Dislocation microstructure after 12% forward shear in the direction SD_1 and 12% reverse shear (resulting $\alpha_P = 6.6\%$) in the direction SD_2 , both parallel to the rolling direction (RD). Partially disintegrated DDWs/CBBs are highlighted by ellipses in (a). On the top of (b) a distorted, initially probably rectangular CB is marked by an ellipse. A γ_1 -grain is shown.

3.7 Plane strain tension-shear

As shown in Fig. 14 for a general γ -grain, in plane-strain tension followed by simple shear, dislocations arrange in a grid-like structure dominated by CBBs. The micrograph shown in Fig. 14 was prepared from a sample subjected to 11 % tension and 35 % shear (resulting $\alpha_P = 30.3\%$). The grid-like structure appears especially in grains with a Schmid factor between 0.25 and 0.5. Here, the Schmid factor refers to the tension phase. More than half of the grains fall into this category, as was determined by EBSD measure-

ments. In many cases the dense dislocation walls run across grain boundaries, even though slight changes in orientation are observed. The schematic (Fig. 14(b)) emphasizes the patterned structure of dense dislocation walls. Several of the DDWs in the horizontal direction have split into double walls (DWs) in Fig. 14(b). The cell-block boundaries parallel to the shear direction in Fig. 14 are former microbands which have cut through the planar dislocation structure from the tensile loading and have evolved into cell-block boundaries with increasing shear load.

(a) Micrograph

(b) Schematic

Fig. 14 Microstructure in a general γ -grain after plane-strain tension to $F_{22} - 1 = 0.11$ in the direction TenD followed by release and reloading in simple shear to $F_{12} = 0.35$ in the direction SD. Note the characteristic grid-like structure formed by CBBs.

In comparison with earlier studies on uniaxial tension to shear tests (e.g. by [1]) it appears that fragmentation in the

uniaxial tension to shear tests is stronger than in the plane strain tension case. This is consistent with the observations made for the differences in monotonic uniaxial tension and plane strain tension in section 3.5.

3.8 Histogrammic analysis

In addition to these investigations, the thickness of DDWs was measured for 20 % ($\alpha_p = 18.2\%$) and 30 % ($\alpha_p = 26.2\%$) uniaxial tension in the RD, 20 % ($\alpha_p = 21.1\%$) plane strain tension in RD as well as 30 % ($\alpha_p = 17.3\%$) and 50 % ($\alpha_p = 28.9\%$) shear in the TD. The DDW thickness was determined only in γ -grains.

Fig. 15 Distribution of thickness of DDWs on the basis of measurements of 174 walls in 11 γ -grains for 20 % ($\alpha_p = 18.2\%$) in uniaxial tension in RD for equidistant histogram bins.

Before taking a picture which was used for the determination of the thickness of DDWs, it was moderately tilted and it was checked that the variation in thickness of the majority of DDWs does not exceed 10 %. Fig. 15 shows the histogram for 20 % uniaxial tension in the RD. The thickness was determined for 174 DDWs in 11 grains. The average (arithmetic mean) thickness of DDWs is 147 nm with a standard deviation of 118 nm. This variation illustrates that at this deformation level the dislocation microstructure is not in the saturated state, and that there are variations due to the specific orientation of a particular grain and its interaction with adjacent grains. At the investigated levels of deformation the determination of the thickness of DDWs is complicated due to the fact that some DDWs are diffuse as mentioned before. This means that dislocation tangles or parts of walls of CBs might be close to the DDW. This is especially the case when DDWs appear to be thicker than 250 nm. This uncertainty in the data is accounted for in the histograms of Fig. 16 by an increase in size of the histogram

bins with increasing wall thickness. Fig. 16(b) (30 % uniaxial tension) and Fig. 16(a) show that the thickness of DDWs decreases with increasing deformation.

Fig. 16 Histograms illustrating the decrease of wall thickness and decrease of variation in wall thickness with increasing deformation as well as the differences in the DDWs for tension and shear, i.e. DDWs in shear are more pronounced leading to a smaller variation. The centers of the leftmost bins are 25 nm and 50 nm.

The average thickness determined on the basis of 84 DDWs in 4 grains is 118 nm with a standard deviation of 53 nm. This illustrates that the DDWs are more pronounced and that there are less fluctuations. The reduction of the thickness of DDWs with increasing deformation seems to be stronger for comparable deformation in simple shear. The average thickness of DDWs was determined as 123 nm with a standard deviation of 84 nm at 30 % ($\alpha_p = 17.5\%$) shear in the TD (Fig. 16(c)). This changes to an average thickness of 68 nm with a standard deviation of 23 nm at 50 % ($\alpha_p = 28.9\%$) shear in the TD (Fig. 16(d)). The thickness of 78 DDWs was determined in 3 grains. The analysis of the histograms shows that at comparable levels of deformation the variations in the thickness of DDWs tend to be smaller in shear than in tension. This confirms the tendencies described in the previous section which were based on optical analysis of micrographs and their schematic counterparts.

4 Analysis and discussion

The interpretation of the investigated TEM images yielded that the average thickness of wall structures (here, we group CWs, DDWs, CBBs together for simplicity) can be used to distinguish the dislocation structures formed during the investigated loading paths: uniaxial tension, plane strain tension, monotonic shear, forward to reverse shear and plane strain tension to shear. Additional criteria are the number of cell structures (CBs and regular cells) per area and the shape of cells and CBs. In particular, a substantial difference between the morphology of the dislocation structure corresponding to tension (plane strain and uniaxial) and simple shear loading was observed. The analysis of the distribution of the thickness of DDWs in a number of grains shows that a tendency that the thickness of DDWs refines with increasing deformation. This was also observed by Li et al. [45] for cold rolling of IF-steel at higher strains (α_p) and by Hughes et al. [51] for nickel. This refinement seems to be more pronounced in the case of simple shear because the rate of decrease in wall thickness per increase of equivalent strain is larger than in uniaxial tension. These statistics show the tendencies of wall thickness evolution. at the relatively small strain range investigated in this study. The cited previous studies focus on a larger strains and only one deformation mode [45, 51]. number of investigated walls. The statistics could be improved by increasing the number of evaluated grains and by focusing on the distribution of structural parameters, e.g. the thickness of DDWs for the different components of the γ -fiber.

In simple shear the morphology of the dislocation structure is also related to the crystal orientation [2]. According to the results of Nesterova et al. [2] the dislocation struc-

tures in stable γ -grains show pronounced DDWs in monotonic shear. There is usually one family of DDWs almost parallel to the rolling, respective, shear direction according to [2]. In the case of general γ -fiber grains, a stronger appearance of two families of DDWs and a stronger inclination of one family of DDWs against the rolling, respective, shear direction is reported. This general tendency can be confirmed. Furthermore, the partial disintegration of the characteristic γ_1 shear dislocation structure during shear to reverse shear is observed. In Fig. 13 the main family of DDWs is almost parallel to the rolling, respective, shear direction. There are some short DDWs inclined 45° towards this direction. Fig. 11 shows that two families of DDWs may also occur in γ_1 grains. Li et al. [45] argued that there is no strict correlation between the local crystallographic orientation and the exact morphology of the dislocation microstructure in IF-steel during rolling.

If the deformation in the transverse direction is neglected in cold rolling, the macroscopic deformation state of rolling and plane strain tension in the rolling direction can be compared. Li et al. [45] investigated the microstructure evolution during cold rolling for rolling reductions between 10 % and 90 % in IF steel. Even though the texture of the initial state of the material used in their study and in the current study varies due to the different processing, the results of Li et al. [45] for the smaller investigated rolling reductions of 10 % ($\alpha_p = 0.12$) and 30 % ($\alpha_p = 0.41$) can be qualitatively compared with the current plane strain tension cases. For 10 % rolling reduction Li et al. [45] report that “one or two well defined sets of dislocation” walls appear and that these are “inclined in the longitudinal plane to RD at an angle of $\approx \pm 40^\circ$ ”. They also observed that “offsets” appear in the dislocation walls which is consistent with the current notion of higher curvature of CBBs in plane strain tension compared to uniaxial tension. As was mentioned in section 3.5 of the current study of plane strain tension, Li et al. [45] observed a tendency towards the formation of only a single set of dislocation walls with increasing deformation during rolling. The results also showed that the CBBs were parallel to a $\{110\}$ slip plane [45] in most cases. The shape of dislocation microstructures in simple shear is more regular, i.e. the CBs are rectangular. The aspect ratio of the two edges of a rectangular CB tends to be close to 1:1. However, the dislocation density in the CBBs in plane strain tension is considerably lower than in simple shear.

A smaller difference in the intensity of fragmentation distinguishes the dislocation structure in plane strain tension and uniaxial tension. The CBs in plane strain tension have a stronger curvature, and the number of cells at the same normal strain is about twice as large in uniaxial tension as compared to plane strain tension. The knowledge of the morphology of dislocation structures for a certain type of deformation is relevant in the context of the “principle

of similitude” discussed in [37]. According to the principle of similitude, a specific material exhibits a specific pattern of low-energy dislocation structures such as the mentioned cell walls, dense dislocation walls and cell block boundaries. Their scaling changes with deformation. This knowledge may be used to analyse whether the deformation in a specimen, e.g. a simple shear test specimen is homogeneous. It may also be investigated whether tension loading is superimposed due to problems with the test setup. Some of the authors of the present study are preparing a work where the homogeneity of the deformation in shear specimen is analysed on the basis of optical strain measurements and TEM-investigations of the dislocation microstructure.

In the case of changes in loading path, the interaction of different dislocation structures formed during different deformation modes leads to changes in the observed patterns. In the case of load reversal, e.g. forward to shear loading, the authors observed a partial disintegration and distortion of cell structures for moderate forward to reverse shear, but hesitate to use the term “dissolution of cells” as Hasegawa et al. [9] used in their work on pure aluminum because this term implies that cells vanish completely. The existence of partially disintegrated and distorted cell structures indicates that the investigated sample has been subjected to a load reversal. Since load reversal leads to the occurrence of the Bauschinger effect [32] and hardening stagnation as Fig. 3 shows, this indicator is an indirect indicator for properties of a sheet forming process. An example for this is the investigation made for the two-stage drawing process of a cup in [23]. Predictions for the punch force, obtained with a model that accounts for the transient hardening behavior after loading path changes, agreed well with the experimental punch force for the second stage. During the second stage the punch force dropped considerably, when parts of the cup experienced a load reversal.

For the case of an orthogonal loading path change, e.g. plane strain tension to shear, the TEM analysis shows that an orthogonal loading path change leads to changes in the dislocation structure. Traces of characteristic elements of the dislocation structure corresponding to monotonic plane strain tension and monotonic simple shear are observed. This results in the “chess board” structure shown in Fig. 14. In the current investigation the dislocation structure after plane strain tension to simple shear was analysed for the first time for interstitial free steel. In previous works on orthogonal loading path changes, similar patterns for uniaxial tension to shear were observed [1, 2, 5]. We support and adopt the explanations for the micromechanical background of cross hardening and the corresponding evolution of the dislocation structure given in [1, 2, 5]. The stress deformation behavior observed for plane strain tension to shear is in qualitative agreement with the one for uniaxial tension to shear reported e.g. in [49]. CBBs associated with the shear load-

ing are observed after plane strain tension to shear loading. We support the idea of Rauch et al. [5] that these CBBs can be associated with glide systems which became active in the shear phase. The CBBs which formed during plane strain tension, represent obstacles to dislocation glide on the glide systems corresponding to shear loading. This results in the increase of the shear stress on the macroscopic scale. The period of hardening stagnation after the loading path change is related to the weakening and partial disintegration of dislocation structures associated with plane strain tension loading. Examples for this are the DWs in Fig. 14 which were formed after splitting of a DDW. This DDW had formed during plane strain tension.

A key idea of this work, i.e. the characterization of the dislocation microstructures for small to moderate strains and different deformation modes strains, is based on many earlier works. In our opinion the first step is the characterization of monotonic deformation paths. Deformation paths involving more than one stage or a combination of base modes might be interpreted as a superposition of already known “base” modes. Important earlier works in this context analyse the principles of scaling and similitude of dislocation structures in single crystals and polycrystalline metals. Several works focus on the analysis of correlation between different microstructural parameters of dislocation structures. Pantleon [50] proposed a statistical model for incidental boundaries (IBs) which relates the square root of the plastic strain α per slip system with the average misorientation angle Θ_{ave} according to

$$\Theta_{ave} = \sqrt{\frac{\pi b P}{d}} \sqrt{\alpha} \quad (1)$$

where b , P and d are the Burgers vector, the immobilization probability and the size d of an IB. Pantleon and Stoyan [4] developed a more elaborate model to establish that there is an anti-correlation between the single misorientation angles of IBs. Their statistical analysis shows that the experimentally observed saturation of the misorientation angle across an increasing number of IBs can be explained by the fact that misorientations in neighboring IBs are of opposite sign. We deduct from these observations that ordering principles or mechanisms exist which result in the formation of dislocation microstructures. Similarly, the morphology of these structures seems to follow certain principles. Hughes and Hansen [51] investigated the evolution of the microstructure during cold rolling in nickel over a strain range up to α_P . The analysis of the average size of IBs d_{ave} and the average misorientation Θ_{ave} shows that the product of these quantities remains constant over this strain range. Similar results were found for α iron by Langford [52]. Hughes and Hansen [51] also found a linear scaling law for the dependence of the average size of GNBs d_{ave}^{GNB} and their thickness d_{ave}^{GNB} . Two

separate (power) scaling laws for the dependence of the average misorientation of GNBs and IBs on the accumulated plastic deformation α_P were identified based on earlier work [53].

The analysis of the morphologies of the dislocation structure and the corresponding statistical analysis of the wall thickness indicate that there is a specific “TEM signature”, a term introduced by a reviewer of a previous version of this work. The term TEM -signature implies that there are unique features of the dislocation structure which can be used to determine which kind of monotonic deformation a material has experienced.

This knowledge may be used in the analysis of deep-drawn parts. The results of simulations with a microstructural model indicate that cross hardening leads to larger deformations due to springback [22]. Wang et al. [22] investigated the ring-opening of split rings cut from deep-drawn cups. A TEM analysis of the dislocation structure may reveal whether loading path changes have led to changes in the dislocation structure. Since there is a correlation between the dislocation microstructure and the stress-deformation behavior, the analysis of the dislocation microstructure may be used to make statements on the deformation history in addition to or independently from simulations.

5 Conclusions

The immediate results of this investigation are:

- There are characteristic features of the dislocation structure which can be used to correlate a loading path to a specific dislocation structure in interstitial free steels.

For the investigated loading paths these characteristics are:

- The characteristic cell shape of simple shear is rectangular with an aspect ratio close to 1:1. The refinement of the wall thickness in simple shear with increasing deformation seems to be greater than in uniaxial tension. There are very few single dislocations inside of cells in simple shear.
- The morphologies of the dislocation structure in simple shear, especially the number of families of DDWs and their orientation, are influenced by the specific crystallographic orientation. The results of the current work indicate that this is a general tendency, but not a strict correlation as reported in previous works.
- At the small strain level investigated the tendency to wall refinement of DDWs can be observed for uniaxial tension and simple shear.
- Additional statistical analysis of a larger number of grains and other structural parameters, e.g. the misorientation and the cell size, will help to characterize the morphology of dislocation structures for the investigated deformation level.

- Cells are polygonal after tension. Most cells are quadrilateral after uniaxial tension. The edges are rather straight in uniaxial tension. In contrast, cells after plane strain tension may have more than four edges. The cell walls show stronger curvature in plane strain tension than in uniaxial tension. The fragmentation is less intense in plane strain tension.
- After loading reversal partial disintegration and distortion of previously formed dislocation structures are observed.
- After orthogonal loading path changes characteristic elements of the dislocation structure belonging to both loading modes are observed. Interaction of different families of dislocations leads to weakening of “old” dislocation structures.

The previous results are relevant in the context of:

- Application of this knowledge to an *a posteriori* analysis of test specimen to verify the homogeneity of the deformation and the degree to which the desired loading path was realized
- Application of this knowledge to an *a posteriori* analysis of sheet metal parts in order to make statements whether the dislocation structure shows indicators of loading path changes in complex parts. If such evidence is found, the transient hardening may have affected the process or the properties of the final part.

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References

1. Nesterova EV, Bacroix B, Teodosiu C (2001) Experimental observation of microstructure evolution under strain-path changes in low-carbon IF steel. *Mater Sci Eng A* 309-310:495-499
2. Nesterova EV, Bacroix B, Teodosiu C (2001) Microstructure and texture evolution under strain-path changes in low-carbon interstitial-free steel. *Metall Mater Trans A* 32A:2527-2538
3. Fernandes JV, Schmitt JH (1983) Dislocation microstructures in steel during deep drawing. *Philos Mag* 48:841-870
4. Pantleon W, Stoyan D (2000) Correlations between disorientations in neighbouring dislocation boundaries. *Acta Mater* 48:3005-3014
5. Rauch EF, Schmitt JH (1989) Dislocation substructures in mild steel deformed in simple shear. *Mater Sci Eng A* 113:441-448
6. Lins JFC, Sandim HRZ, Kestenbach HJ (2007) Microstructural and textural characterization of a hot-rolled IF steel. *J* 42:6572-7
7. Wilson DV, Bate PS (1994) Influences of cell walls and grain boundaries on transient responses of an IF steel to changes in strain path. *Acta Metall Mater* 42:1099-1111
8. Vincze G, Rauch EF, Gracio JJ, Barlat F, Lopes AB (2005) A comparison of the mechanical behaviour of an AA1050 and a low carbon steel deformed upon strain reversal. *Acta Mater* 53:1005-1013
9. Hasegawa T, Yakou T, Karashima S (1975) Deformation behaviour and dislocation structures upon stress reversal in polycrystalline aluminium. *Mater Sci Eng* 20:267-276
10. Bouvier S, Alves J, Oliveira M, Menezes L (2005) Modelling of anisotropic work-hardening behaviour of metallic materials subjected to strain-path changes. *Comput Mater Sci* 32:301-315
11. Figueiredo R, Corrêa E, Monteiro W, Aguilar M, Cetlin P (2010) The effect of cyclic bending on the mechanical properties and dislocation structures of drawn steel bars. *J Mater Sci* 45:804-810
12. Ding X, He G, Chen C (2010) Study on the dislocation sub-structures of Al-Mg-Si alloys fatigued under non-proportional loadings. *J Mater Sci* 45:4046-4053
13. Landau P, Shneck R, Makov G, Venkert A (2007) Microstructure evolution in deformed copper. *J Mater Sci* 42:9775-9782
14. Thuillier S, Rauch EF (1994) Development of microbands in mild steel during cross loading. *Acta Metall Mater* 42:1973-1983
15. Teodosiu C, Hu Z (1998). Microstructure in the continuum modelling of plastic anisotropy. *Proc 19th Risø Inter Symp Mater Sci: Model Struct Mech Mater Mic Prod, Risø Nat Lab, Roskilde, Denmark*, pp 149-68
16. Noman M, Clausmeyer T, Barthel C, Svendsen B, Huétink J, van Riel M (2010) Experimental characterization and modeling of the hardening behavior of the sheet steel LH800. *Mater Sci Eng A* 527:2515-2526
17. Teodosiu C, Hu Z (1995) Evolution of the intragranular microstructure at moderate and large strains: modelling and computational significance, In: Shen SF, Dawson PR (ed), *Sim Mater Process: Theory, Methods and Applications*. Balkema, Rotterdam, pp 173-182
18. Wang J, Levkovitch V, Reusch F, Svendsen B, Huétink J, van Riel M (2008) On the modeling of hardening in metals during non-proportional loading. *Int J Plast*

- 24:1039-1070
19. Peeters B, Kalidindi SR, Teodosiu C, van Houtte P, Aernoudt E (2002) A theoretical investigation of the influence of dislocation sheets on evolution of yield surfaces in single-phase b.c.c. polycrystals. *J Mech Phys Solid* 50:783-807
20. Holmedal B, van Houtte P, An Y (2008) A crystal plasticity model for strain-path changes in metals. *Int J Plast* 24:1360-1379
21. Beyerlein I, Alexander D, Tomé C (2007) Plastic anisotropy in aluminum and copper pre-strained by equal channel angular extrusion. *J Mater Sci* 42:1733-1750
22. Wang J, Levkovitch V, Svendsen B (2006) Modeling and simulation of directional hardening in metals during non-proportional loading. *J Mater Process Technol* 177:430-432
23. Thuillier S, Manach PY, Menezes LF (2010) Occurrence of strain path changes in a two-stage deep drawing process. *J Mater Process Technol* 210:226-232
24. van Riel M, van den Boogaard AH (2007) Stress-strain responses for continuous orthogonal strain path changes with increasing sharpness. *Scr Mater* 57:381-384
25. Cao J, Shi MF, Stoughton TB, Wang CT, Zhang L (2005) Proc NUMISHEET 2005 - The Numisheet 2005 Benchmark Study, Part B, Detroit, MI, USA, Am Inst Phys 778:881-1190
26. DIN EN 10130 (2006) Cold rolled low carbon steel flat products for cold forming Technical delivery conditions. Techn rep DIN
27. Clausmeyer T, van den Boogaard AH, Noman M, Gershteyn G, Schaper M, Svendsen B, Bargmann S (2011) Phenomenological modeling of anisotropy induced by evolution of the dislocation structure on the macroscopic and microscopic scale. *Int J Mater Form* 4:141-154
28. van Riel M (2009) Strain path dependency in sheet metal - experiments and models. Dissertation, Universiteit Twente
29. Kuwabara T (2007) Advances in experiments on metal sheets and tubes in support of constitutive modeling and forming simulations. *Int J Plast* 23:385-419
30. Hu Z (1994) Work-hardening behavior of mild steel under cyclic deformation at finite strains. *Acta Metall Mater* 42:3481-3451
31. Bouvier S, Teodosiu C, Haddadi H, Tabacaru V (2003) Anisotropic work-hardening behaviour of structural steels and aluminium alloys at large strains. *J Phys IV France* 105:215-222
32. Bauschinger J (1881) Über die Veränderung der Elastizitätsgrenze und des Elastizitätsmoduls verschiedener Metalle. *Zivilingenieur* 21:289-348
33. Hielscher R, Schaeben H (2008) A novel pole figure inversion method: specification of the MTEX algorithm. *J Appl Crystallogr* 41:1024-1037
34. Bacroix B, Hu Z (1995) Texture evolution induced by strain path changes in low carbon steel sheets. *Metall Mater Trans A* 26:601-613
35. Hughes DA, Hansen N (1991). Microstructural evolution in nickel during rolling and torsion. *Mater Sci Technol* 7:544-553
36. Kuhlmann-Wilsdorf D, Hansen N (1991) Geometrically necessary, incidental and subgrain boundaries. *Scr Metall Mater* 25:1557-15622
37. Kuhlmann-Wilsdorf D (1989) Theory of plastic deformation: - properties of low energy dislocation structures. *Mater Sci Eng A* 113:1-41
38. Juda U, Frank-Rotsch C, Rudolph P (2008) Analysis of dislocation cell patterns in as-grown compound materials (GaAs, CaF₂). *J Mater Sci* 19:342-346
39. Kurzydłowski KJ (1995) The quantitative description of the microstructure of materials. CRC Press, London
40. Rasband WS (2004) ImageJ. National Institutes of Health, Bethesda, MD, USA, <http://rsb.info.nih.gov/ij/>. Accessed July 12 2012
41. Gonzalez RC, Woods RE, Eddins SL (2008) Digital image processing using MATLAB®. Gatesmark Publishing
42. Bailey JE, Hirsch PB (1960) The dislocation distribution, flow stress, and stored energy in cold-worked polycrystalline silver. *Philos Mag* 5:485-497
43. Stremel MA, Belyakov BG (1968) Possibilities of electron microscopic studies of the dislocation density. *Phys Metal Metallogr* 25:140-151
44. Rybin VV (1986) Severe Plastic Deformations and Fracture of Metals. Metallurgiya, Moscow
45. Li BL, Godfrey A, Meng QC, Liu Q, Hansen N (2004) Microstructural evolution of IF-steel during cold rolling. *Acta Mater* 52:1069-1081
46. Keh AS, Spitzig WA, Nakada Y (1971) Dislocations structures in deformed iron single crystals. *Philos Mag* 23:829-846
47. Spitzig WA, Keh AS (1971) Orientations and temperature dependence of slip in iron single crystals. *Metall Trans* 1:2751-2757
48. Böhm H (1968) Einführung in die Metallkunde. Verlag Bibliographisches Institut AG, Mannheim
49. Haddadi H, Bouvier S, Banu M, Maier C, Teodosiu C (2006) Towards an accurate description of the anisotropic behaviour of sheet metals under large plastic deformations: modelling, numerical analysis and identification. *Int J Plast* 22:2226-2271
50. Pantleon W (1998) On the statistical origin of disorientations in dislocation structures. *Acta Mater* 46:451-456

51. Hughes DA, Hansen N (2000) Microstructure and strength of nickel at large strains. *Acta Mater* 48:2985-3004
52. Langford G, Cohen M (1975) Microstructural analysis by high-voltage electron diffraction of severely drawn iron wires. *Metall Mater Trans A* 6:901-910
53. Godfrey A, Hughes DA (2000) Scaling of the spacing of deformation induced dislocation boundaries. *Acta Mater* 48:1897-1905