



# Experimental mesoscopic investigation of the local cyclic plasticity of a non-oriented electrical steel

Jeremie Bouquerel, C. Schayes, Jean-Bernard Vogt

## ► To cite this version:

Jeremie Bouquerel, C. Schayes, Jean-Bernard Vogt. Experimental mesoscopic investigation of the local cyclic plasticity of a non-oriented electrical steel. *Materials Science and Engineering: A*, 2021, *Materials Science and Engineering: A*, 820, pp.141454. 10.1016/j.msea.2021.141454 . hal-03259323

**HAL Id: hal-03259323**

**<https://hal.univ-lille.fr/hal-03259323>**

Submitted on 13 Jun 2023

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial 4.0 International License

# Experimental mesoscopic investigation of the local cyclic plasticity of a non-oriented electrical steel

J. Bouquerel\*, C. Schayes, J.B. Vogt

Univ. Lille, CNRS, INRAE, Centrale Lille, UMR 8207 -

UMET - Unité Matériaux et Transformations, F-59000 Lille, France

\*Corresponding author : [jeremie.bouquerel@centralelille.fr](mailto:jeremie.bouquerel@centralelille.fr)

## Abstract

Complementary mesoscopic investigations have been carried out on a notched specimen, made of Fe-3%Si steel and submitted to fatigue loading. Load control fatigue testing was conducted to ensure localised cyclic deformation and the resulting displacement fields were measured by means of Digital Image Correlation (DIC). Electron Channelling Contrast Imaging (ECCI) and Electron Back-Scatter Diffraction (EBSD) complete the DIC data by giving a different view of the local plasticity, as they image the dislocation structure and map their related characteristics, such as misorientation.

Hence, two misorientation based criteria, namely Grain Reference Orientation Deviation (GROD) and Grain Orientation Spread (GOS) were considered for EBSD analysis. Based on a previous study, a  $\langle \text{GOS} \rangle - \Delta \varepsilon_t$  abacus has been coupled with a  $\langle \text{GOS} \rangle$  - DIC abacus in order to evaluate locally the encountered strain field and link this latter to an equivalent total strain variation. The obtained results have been discussed from the evolution of the dislocations structures, as observed by ECCI.

This combination of mesoscopic techniques clearly emphasizes the local deformation by means of both qualitative and quantitative measurements. On one hand, DIC and EBSD measurements indicate the presence of strain localisation which can be correlated to a certain level of cyclic strain amplitude. On the other hand, the variation of the strain levels and its distribution when moving away from the notch are confirmed by the dislocation structures evolution seen by ECCI. Such

structures vary from cells, vein and channels and entangled dislocations regions as the analysed areas go away from the notch. At a local scale, the DIC measurements behave as numerous virtual extensometers while its combination with EBSD and ECCI data confirms the correlation with the accumulated cyclic strain.

## **1. Introduction**

Nowadays, the knowledge and the understanding of the plasticity phenomena remain fantastic challenges for both engineers and researchers. On one hand, it allows the design of materials and/or components that exhibit superior mechanical properties by optimising their microstructure and by improving thermomechanical treatments to which those latter are submitted. On the other hand, it allows a lifetime optimisation as materials may undergo various types of constraints when in operating conditions.

One of the constraints which has a high occurrence concerns the fatigue loading, which can be related to expected or unexpected in-service conditions. Indeed, fatigue corresponds to the progressive and structural damage that occurs when a material is subjected to cyclic loading. It is a major cause of component failure in many industrial domains such as construction [1,2], energy [3–5] and transportation [6–10].

The Fe-3%Si steel (M330-35A) is of prime interest in regards with the industrial sectors mentioned above. As it belongs to the non-oriented electrical steels, it is frequently used for its electrotechnical properties obtained by a well-controlled combination of chemical composition, grain-based features (morphologic and crystallographic texture) and sheet thickness (350  $\mu\text{m}$ ). It is then particularly adapted to rotating machines such as alternators.

This steel and more generally the iron-silicon electrical steels have been specifically optimised for their electrical properties and more precisely for the prediction of core losses to improve electrical machine design [11–14], but less their mechanical properties. Nevertheless, the rotor design

improvement requires now to clearly know their mechanical properties and more precisely their fatigue properties.

Within the last decades, metallurgists and mechanical engineers have developed robust characterisation tools to characterise the plastic deformation that occurs during fatigue. Although the synergies and tools were initially different, the frontier between their approaches become more and more narrow. Indeed, metallurgists pay more attention to microstructural developments ranging from more or less complex dislocation interactions to more mesoscopic misorientation build-up. To their side, the mechanical community evaluated deformation in terms of stresses and strains and part of them are focusing on microstructural impact through micromechanics.

Several interesting tools may then be considered for investigations at the mesoscopic and macroscopic scales. Electron Back-Scatter Diffraction (EBSD) and Electron Contrast Channelling Imaging (ECCI) bring valuable information regarding deformation as it can be viewed from microstructural evolution. Digital Image Correlation (DIC) gives quantitative data regarding the strain and stress field at various scales. Finally, simulation based on Finite Element Modelling (FEM), arises to predict or validate materials and component behaviours.

All those techniques take benefits from a constant development brought from their respective community, which give a more precise ability for the plastic deformation analysis [15–24]. When regarding their applications to fatigue behaviour, those approaches are often used separately [25–27], although few recent works tried to perform some correlation [24,28–31]. However, only a fragmentary view of the material characteristics is treated in order to describe mechanisms related to crack initiation or first steps of crack propagation by focusing on.

The purpose of the present work is somehow different. Two previous studies performed on the fatigue behaviour of the M330-35A steel point out the ability of EBSD and ECCI to give mesoscopic details allowing the explanation of the stress response of the material to strain cycling [32,33]. It was also shown that, depending on the applied strain, this steel exhibits behaviours corresponding to low

temperature and high temperature regimes. Moreover, EBSD based criteria pointed out the associated strain localisation that leads to crack initiation. In the present study, those results are used as a kind of abacus in order to estimate the fatigue strain encountered during the cyclic loading of a notched sample. The comparison between the SEM based analysis and the DIC measurements validates the previous approach on a sample with a more complex shape, for which both stress and strain distribution is heterogeneous. Mesoscopic DIC measurements gives a large amount of local virtual extensometers for the different areas of the sample, while EBSD/ECCI confirm the encountered strain levels assess the presence of dislocations cells.

## 2. Material and experimental procedure

### 2.1. Material

The material considered in this study is the M330-35A of the EN10106 classification (or graded as 36F185 according to ASTM A677). This corresponds to a non-oriented silicon steel with the chemical composition given in Table 1.

**Table 1: Chemical composition of the M330-35A steel (wt%)**

Elements	C	Mn	P	Si	S	Al	Fe
wt%	0.003	0.109	0.011	2.884	0.004	0.448	Bal.

The material has been provided in thin sheet, 350  $\mu\text{m}$  in thickness, but no details regarding the rolling conditions have been given by the supplier. It exhibits a ferritic structure (Figure 1) with a wide grain size distribution [32–34]. The average grain size determined by the intercept method is 75  $\mu\text{m}$ . The sheet thickness corresponds therefore to a thickness of 4 to 5 grains. The grains are not randomly oriented but are assumed to show a  $[1\ 1\ 1] // \text{ND}$  fibres, resulting from recrystallization as suggests the literature on iron silicon steels [35,36]. The ideal texture for non-oriented silicon steel is  $(001) [\text{uv}0]$  where each grain has two  $\langle 1\ 0\ 0 \rangle$  directions in the sheet plane, and the properties are nearly isotropic but no industrial process has been developed to obtain this ideal structure yet [37].

M330-35A is a soft and ductile material with a Young modulus  $E=186$  GPa and an elongation to fracture  $A=28\%$ .

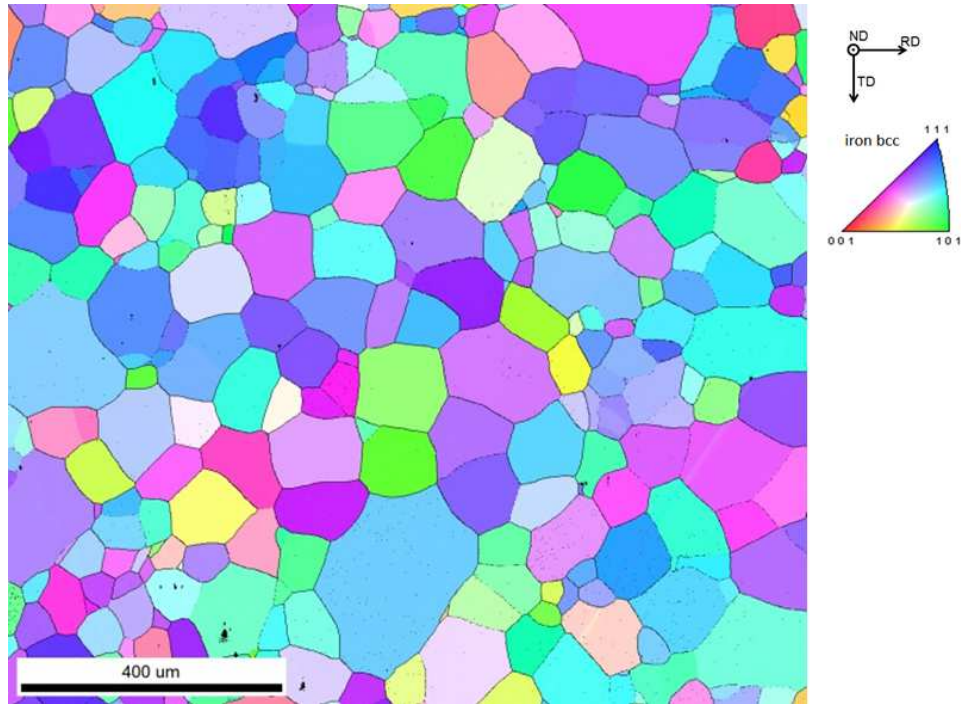


Figure 1: Microstructure of the M330-35A steel

## 2.2. Fatigue tests

In the previous studies, the main idea was to perform conventional Low Cycle Fatigue (LCF) tests on smooth specimen to understand the behaviour of the M330-35A steel. In the present case, the purpose is double. By making a notch in a flat sample, this reproduces a shape feature of a real component and ensure stress concentrators which may modify the local strain levels.

The LCF tests were carried out on a MTS servo-hydraulic machine on the same shape used in the previous investigations unlike the introduction of a rounded notch at the mid place of an edge of the gage length. With this specimen shape (cf. figure 2.a), it is not relevant to perform strain controlled LCF tests. Hence, a load control ranging from 0 to 700 N in a push-pull mode and with a triangular wave form was used instead.

### 2.3. Digital Image Correlation (DIC)

The DIC analysis, which consists in an advanced non-contact optical technique for displacement measurement, has been performed on a 2D system from GOM system (Gesellschaft für Optische Messtechnik). The considered system is constituted of a CCD camera, fitted with a 100mm lens, has a resolution of 2448\*2050 pixels and a maximum frame rate of 15Hz. The post-processing of the data was performed with ARAMIS® v6.3.0. software (GOM mbH, Braunschweig, Germany).

To conduct the DIC analyses, the notched specimen was first mechanically polished up to 3µm. Then, to obtain a speckle surface, a white antireflection paint was applied uniformly on the surface. Once it was dry, a graphite-based spray is applied in order to obtain correct grey distribution from the random repartition of black dots on the surface (Figure 2b). This preparation is crucial in order to have enough contrast and pattern features that allows to perform comparative analysis. [38,39]

The sample was then put into the fatigue testing machine and the camera was placed in front of the specimen at 90° to its surface (Figure 2c).

Image acquisition was performed before the fatigue test and during the cycle number N=1, 10, 11, 50, 100, 500, 1000 and 2000. The framerate acquisition, coupled with a strobe effect, allows to acquire around 30 snapshots per cycle.

Thanks to those patterns acquisition, the residual strain field in the unloading state has been evaluated by comparing the DIC data taken at the minimum stress at each fatigue cycles in comparison to the reference image taken before the fatigue test.

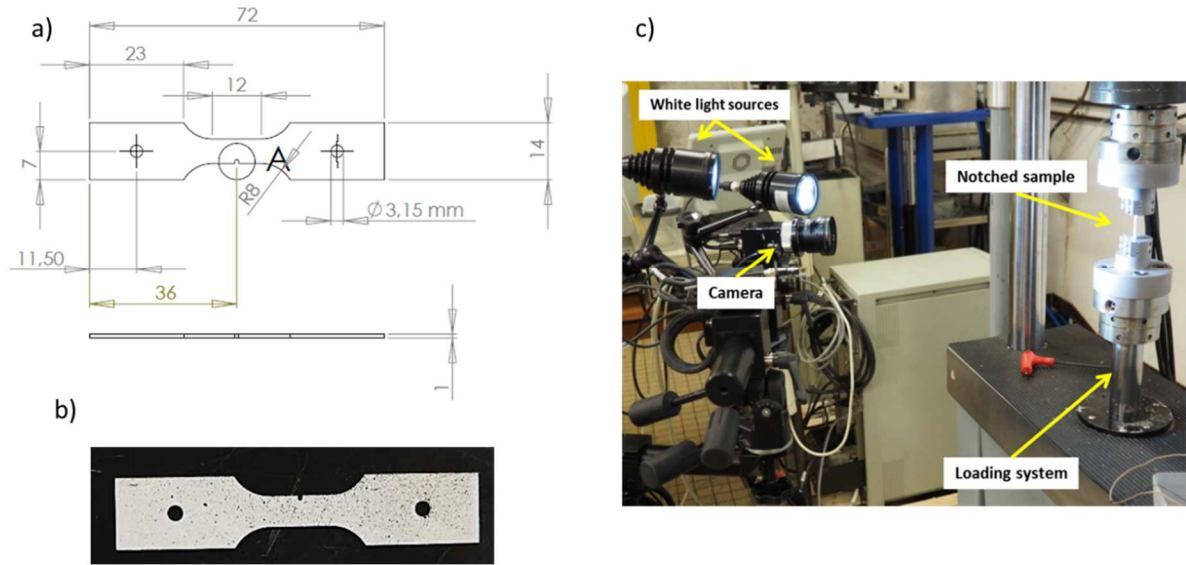


Figure 2: (a) Dimensions of the notched sample, (b) Speckle surface of a notched sample and (c) experimental set-up

## 2.4. Electron Back Scatter Diffraction

EBSD analysis have been carried out on a FEI Quanta 400 scanning electron microscope fitted with an Oxford Instruments EDS/EBSD system. The purpose was to obtain EBSD pattern which processing provides localisation of the strain at a mesoscopic scale.

A Nordlys CCD camera was used for pattern acquisition. Data were recorded at 1  $\mu\text{m}$  step size using Oxford Instruments Aztec software. The analyses were carried out using both Oxford Instruments Channel 5 and TSL OIM 7 commercial software. By choosing the highest possible image resolution for pattern processing and by optimising the Hough transform parameters, an angular resolution of about  $0.3^\circ$  has been measured. As the grain size of the studied steel is relatively large, the considered step size for acquisition (1  $\mu\text{m}$ ), has been chosen according to the Chen *et al.* optimization method [40]. The observations were made on zones of 800  $\mu\text{m}$  x 450  $\mu\text{m}$  in the centre of the useful area of the fatigue samples.

The local strain levels of the Fe-3Si steel were considered through the Image Quality (IQ) of the pattern and the study of local misorientation. As kernel and grain based approaches have been fully investigated in a previous work [32], two criteria are considered here, respectively GROD and GOS as



they provide more evidences of the local strain. Those criteria, which belong to the grain-based approach, are detailed hereafter.

Grain Reference Orientation Deviation (GROD) is based on the misorientation between a reference point of that grain and the other points. The reference point can be the mean misorientation of the grain or the point of the grain where the KAM is the lowest. Note that the KAM value is defined as the mean value of misorientation a pixel and its neighbours providing that misorientation does not exceed 5°, which should correspond to a Low Angle Grain Boundary (LAGB). It is defined by equation (1-2).

$$GROD_i = \omega_{ik} \quad (1)$$

With  $\omega_{ik}$  the deviation angle between orientation of pixel  $i$  and reference orientation of grain  $k$ . This GROD criterion is sensitive to the orientation heterogeneities that evolve during deformation.

Grain Orientation Spread (GOS) is based on the misorientation averaged on the whole grain and is defined by equation (5-4). In this mode each point in the grain is shaded with the same colour in the maps.

$$GOS_k = \frac{1}{M} \sum_{i=1}^M GROD_i \quad (2)$$

With  $M$  the numbers of pixel within the grain  $k$ .

Here, the GOS criterion indicates the grains, as defined by orientation parameters, for which the orientation heterogeneities are the most important.

## 3. Results and discussion

### 3.1. DIC displacement field measurements

As mention previously, the residual displacement field in the deformed image was estimated with the Aramis software by comparison with the initial image of the non-deformed state. From this first set of data, the strain along the Y axis (i.e. loading axis),  $\varepsilon_y$ , and shear strain,  $\varepsilon_{xy}$  were then calculated

at each stage. The strain distributions at  $F_{\max}$  are shown in Figure 3 as a function of the number of cycles. Strain was measured with an error of 0.1%.

These results highlight the longitudinal strain localisation at the notch root and the shear bands and the shear strain in the shear bands. Due to the subset size (9 pixels), which corresponds to a DIC spatial resolution of 210  $\mu\text{m}$ , the strain distribution in the exact vicinity of the notch cannot be precisely evaluated.

The evolution of the longitudinal strain  $\epsilon_y$  at the point where the strain is the maximum at the notch root was also evaluated and reported in Figure 4a and 4b.

Figure 4a shows that the cyclic evolution of the longitudinal strain is in agreement with the cyclic load, the strain increases when the force increases. Moreover, the maximum strain increases with the number of cycles up to  $N=500$  cycles. From this number of cycles, the strain evolution is in a stabilised regime where no more plastic straining occurs as shown in the hysteresis curves (figure 4b).

Note that the presence of a notch induces strain localisation and a behaviour different from the one observed on smooth samples. Indeed, on a smooth specimen, until the propagation of the main crack, the strain partition is more or less homogeneous. The resulting measurement by DIC would be similar to the one obtained with a classical extensometer. In the current case, the most relevant comparison with smooth samples is to consider an incremental step test method, which is generally used to obtain a cyclic stress-strain curve. This test was performed under load control mode and the strain was measured by an extensometer. Once the strain was stabilised, the step was finished and the load was increased for the following loading step. The advantage of this test is that it needs only one sample. However, it is important to specify a parameter for the end of a step which is characterised by a stabilisation of the deformation during cycling. It is considered that the stabilisation is reached when the difference between two cycles is inferior or equal to  $10^{-4}/\text{cycle}$  as it is shown in Figure 5a.

Table 1 summaries the results for the considered Fe-3Si steel. The obtained cyclic stress-strain curve is represented in Figure 5b and compared with the monotonic tensile curve. A cyclic hardening is observed and for a same stress, the strain is more important in monotonic loading than in cyclic loading.

Hence, the value of 3% measured by DIC at the notch root after 2000 cycles results from the cycling at a positive mean stress. When a material is subjected to such condition of loading, it is observed that the hysteresis loops progressively shift toward positive value of strain and a steady state can be observed or not. The latter case refers to the ratcheting effect and the total deformation continuously increases [41]. When a steady state is observed, the total strain remains constant and the stress-strain evolution evolve either in an elastic way or with plasticity. Figure 4b suggests that a steady state has occurred, the loops do not shift anymore with increasing number of cycles since 500 cycles and very small plastic strain can be measured on the hysteresis loops. Therefore, the strain value of 3 % results from an accumulation of cycles. Nevertheless, one may not exclude that the strain at the notch tip resulting from each type of loading would be different from an equivalent static loading. Indeed, the strain assessed by the DIC technique is only based on the processing of displacement field. However, the microstructure behind will be different since the plasticity at the notch root would activate differently the dislocation displacement.

Note that ratcheting effect also occurs during the incremental step test method. Moreover, the behaviour of the steel in the localised deformation areas (as observed visually on Figure 4a) corresponds to zones for which the material response is similar to regimes  $\sigma_{ys} < \sigma_{max} < 1.2 \sigma_{ys}$ . Nevertheless, due to stress concentration effect, the associated strain amplitude is more pronounced.

Then, in order to correlate these results in a more precise way, both EBSD and ECCI techniques are necessary to be used to complete the DIC measurements otherwise incorrect plasticity mechanisms may be proposed.

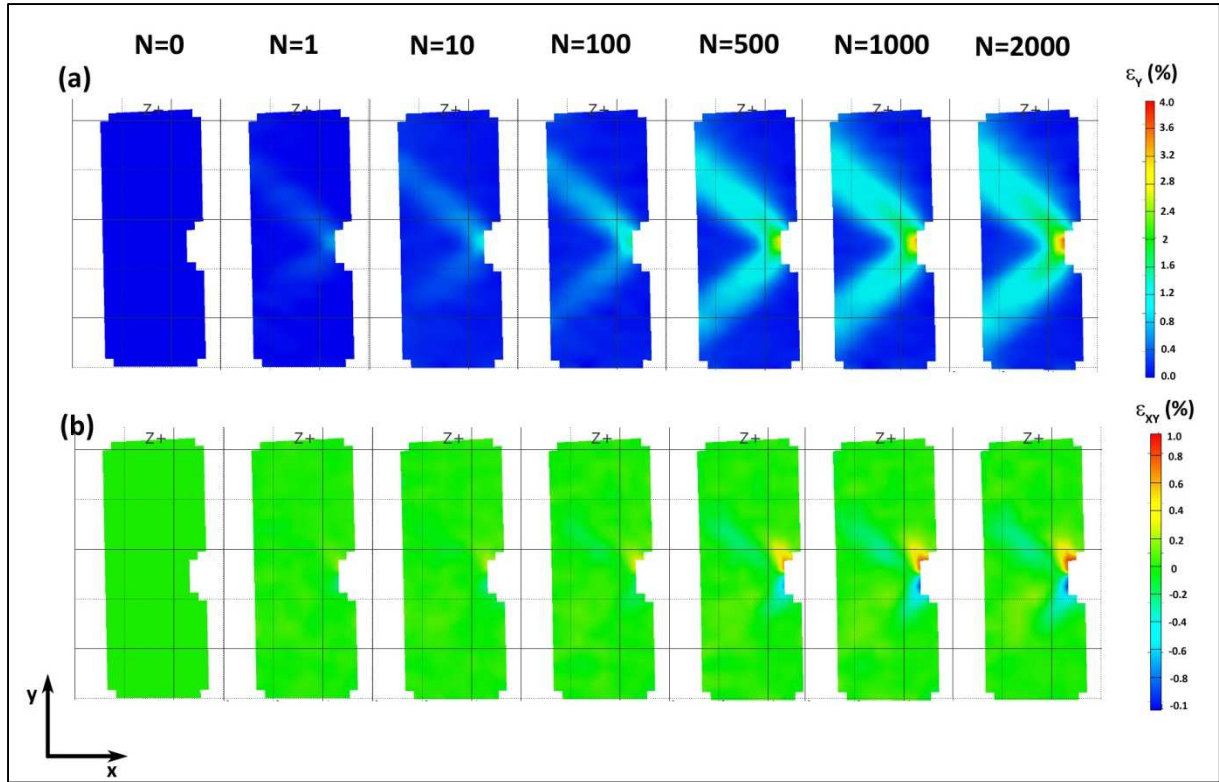


Figure 3: (a) Longitudinal strain,  $\epsilon_y$ , and (b) shear strain,  $\epsilon_{xy}$ , distribution estimation by DIC of a notched sample subjected to cyclic loading at  $F_{max}$  for cycle number  $N=0, 1, 10, 100, 500, 1000$  and  $2000$

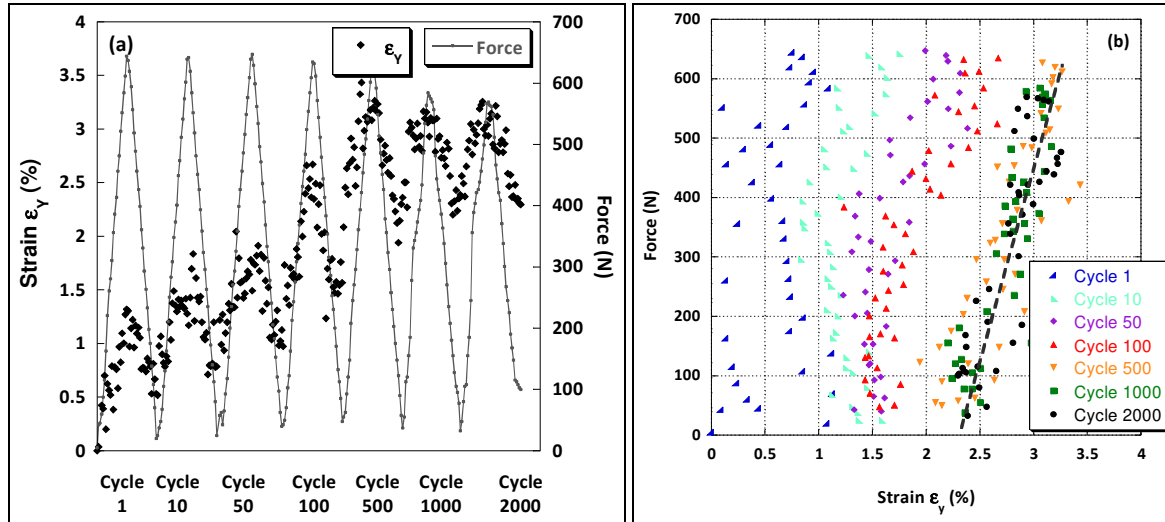
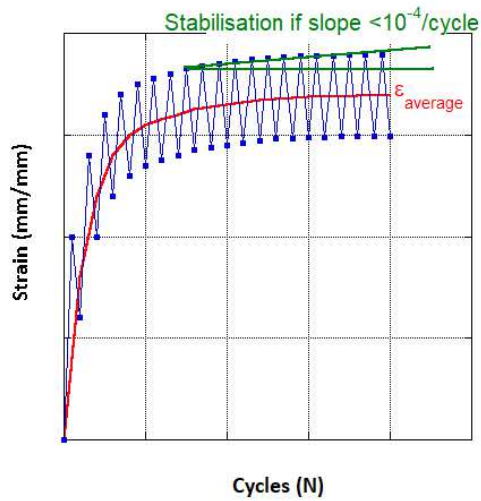
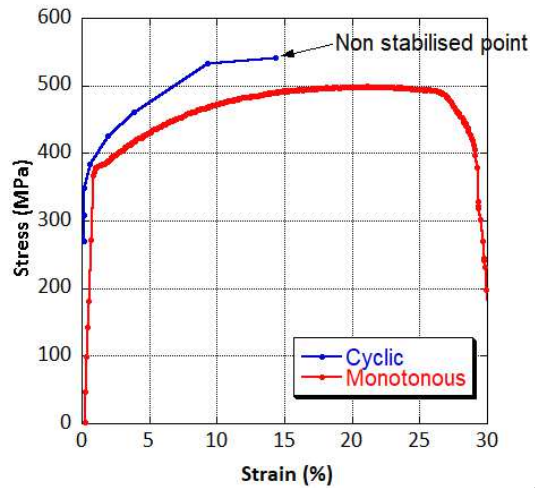


Figure 4: (a) Evolution of the longitudinal strain  $\epsilon_y$  at the notch root and (b) hysteresis curves as a function of the cycle number



(a)



(b)

Figure 5: (a) Stabilisation criteria of a step (b) Cyclic vs monotonic stress-strain curve of the Fe-3Si steel

Table 1: Summary of the fatigue test by steps

$\sigma_{\max}$	Material response			Strain (stabilised regime)			Number of cycles to reach the stabilisation criteria
	$\sigma_{\max}$ (MPa)	$\sigma_{\min}$ (MPa)	$\sigma_a$ (MPa)	$\epsilon_{\max}$ (%)	$\epsilon_{\min}$ (%)	$\Delta\epsilon_t$ (%)	
0.7 $\sigma_{ys}$	253	4.4	129.02	0.126	0.008	0.118	100
0.8 $\sigma_{ys}$	292	3.6	148	0.146	0.008	0.138	100
0.9 $\sigma_{ys}$	330	2	166.42	0.175	0.018	0.158	200
$\sigma_{ys}$	364	3.2	138.62	0.622	0.46	0.162	4000
1.1 $\sigma_{ys}$	403	1.2	202.22	1.89	1.69	0.2	4000
1.2 $\sigma_{ys}$	437	4.8	221.03	3.83	3.59	0.24	5000
1.3 $\sigma_{ys}$	495	29.6	262.4	9.31	9.02	0.29	500
1.4 $\sigma_{ys}$	508	7.2	257.83	15	14.7	0.34	Non stabilised

### 3.2. EBSD analysis

To highlight the ability of the EBSD tool to indicate the strain localisation effect, EBSD analyses were carried out on the notched sample used for the study of strain distribution by DIC, at N=2000 cycles. Figure 6 indicates the areas considered for the EBSD analysis on the normal strain distribution map measured by DIC. The two considered zones are, for the first one, just behind the notch (zone 1) and in the vicinity of a shear band (zone 2).

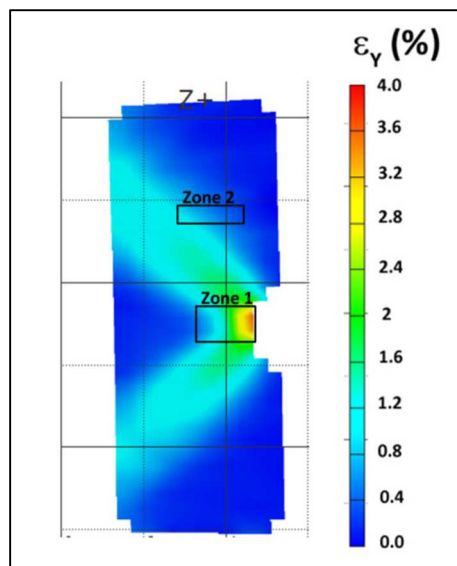


Figure 6: Localisation of complementary EBSD analyses

In order to have an overview of these broad areas, several maps of  $800 \times 450 \mu\text{m}$  were recorded and stitched to each other in order to form a global map of  $1570 \times 750 \mu\text{m}^2$  (279 grains) for zone 1 and of  $3000 \times 815 \mu\text{m}^2$  (483 grains) for zone 2. The corresponding colour coded IPF, GROD and GOS maps of zone 1 and zone 2 are shown in Figure 7 and Figure 8 respectively.

Both IPF, GROD and GOS maps of the zone 1 (figure 7) show orientation heterogeneities close to the notch. For the IPF map (figure 7a), these heterogeneities correspond to local variation of the colour contrast. The GROD map (figure 7b) shows that close to the notch (on the right) warmer colours are obtained, which is the sign of higher misorientation (up to  $8^\circ$ ) and strong localisation of orientation

251 heterogeneities at the grain boundaries. Further away from the notch, grain interiors become almost  
252 free from misorientation gradients while those latter remain localised at the grain boundaries  
253 vicinity.

254 The GOS map (figure 7c) attributes a grain colour that depends on the presence of misorientation  
255 gradients within the grain. A gradient colour mapping is introduced: as the misorientation gradients  
256 inside the grain become stronger, the colour evaluates from cold (blue, 0°) to warm (red, 5°) colours.

257 The EBSD investigation of the zone 1 shows that the grains become less misoriented away from the  
258 notch. This observation is consistent with the strain localisation in this area. Indeed, strain is  
259 correlated with the presence of both statistically stored dislocations (SSD) and geometrically  
260 necessary dislocations (GND), and grain misorientation in deformed areas are inherited from their  
261 presence. Moreover, the misorientation gradients observed inside the grain are consequences of  
262 different dislocations signatures in the grain interior (vein structures) and at the grain boundary (wall  
263 structures) [42].

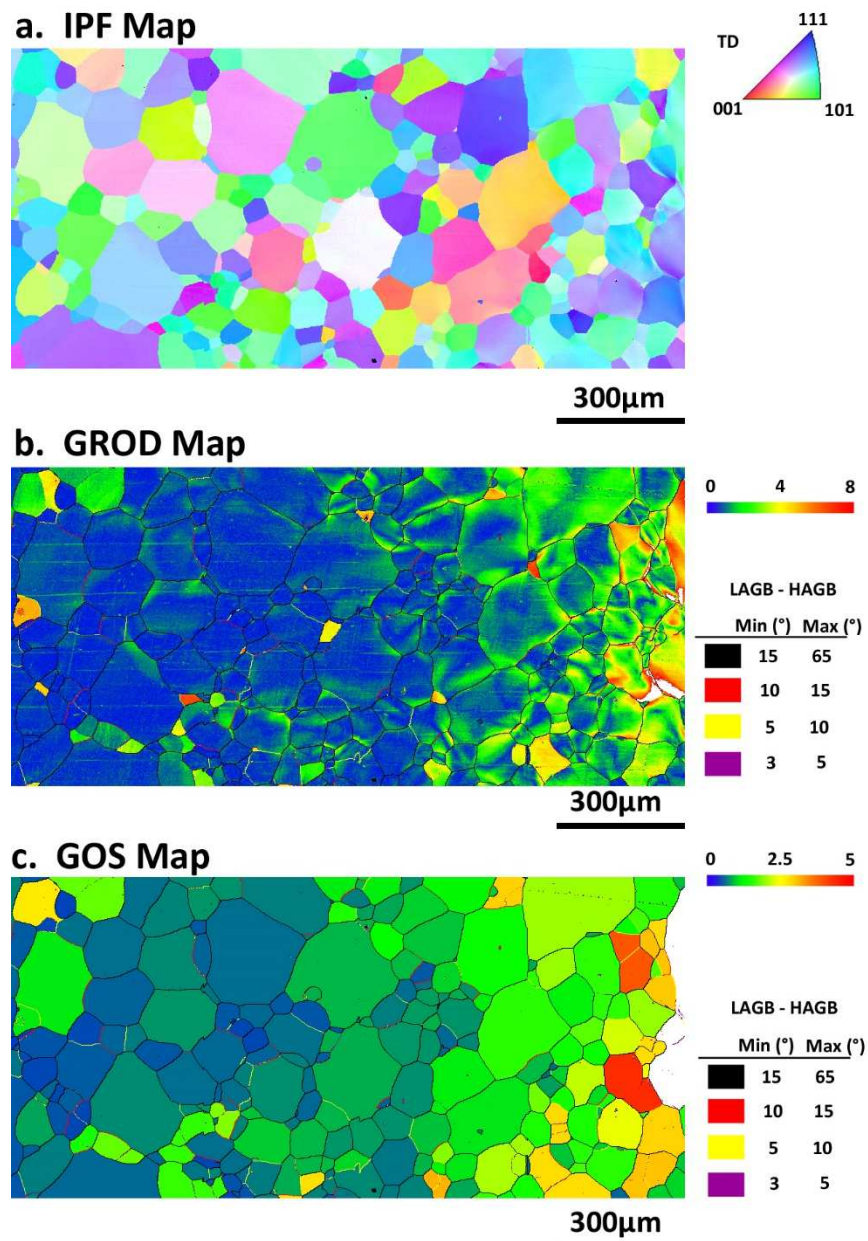
264 For further examinations and correlation with microstructure details and measured DIC values, the  
265 global GOS map was divided in 4 subsets, defined with the black dot square areas of GOS map (figure  
266 7c). The mean GOS, calculated in these subsets, are the following:  $GOS_{mean,1}=1.55$ ,  $GOS_{mean,2}=0.89$ ,  
267  $GOS_{mean,3}=0.69$ ,  $GOS_{mean,4}=0.6$ .

268 Regarding the zone 2, on the left of the map (figure 8b), there is a slight misorientation gradient  
269 within the grains, associated with a localisation at the grain boundary vicinity. Note that, some grains  
270 exhibit higher misorientations levels. The trend is different on the right side of the map, for which  
271 almost all the grains exhibit no misorientation gradient. This observation is confirmed by the GOS  
272 map (figure 8c), which show a higher amount of grain free from misorientation gradient on the on  
273 the right of the map. This observation is coherent with the DIC map (figure 6). Indeed, the presence  
274 of the shear band which induces higher strain in the left part of the zone 2.

275 In this zone, the mean GOS (calculated from the black dot square area of the GOS map in figure 8) is  
 276 0.62 and corresponds to the value  $GOS_{mean,4}$  measured previously.

277 This quick comparison between, *i.* the DIC strain map of the notch sample at N=2000 cycles and *ii.*  
 278 the localisation of the areas on which EBSD analysis has been carried out, confirms the ability of EBSD  
 279 to emphasize the plastic strain localisation.

280



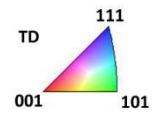
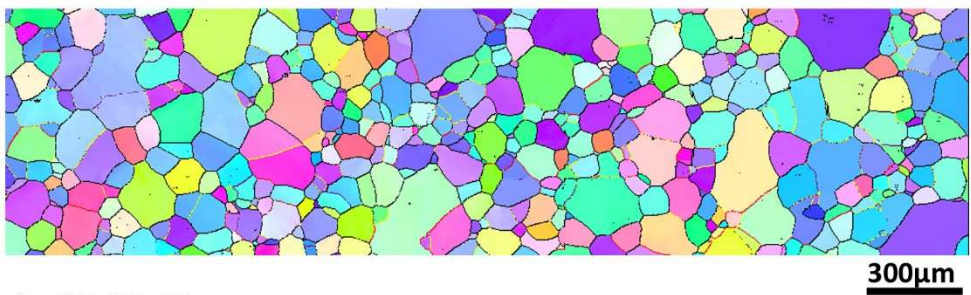
281

282 Figure 7: (a) IPF in TD direction, (b) GROD and (c) GOS maps of the Zone 1 of the sample (notch on the right)

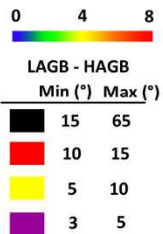
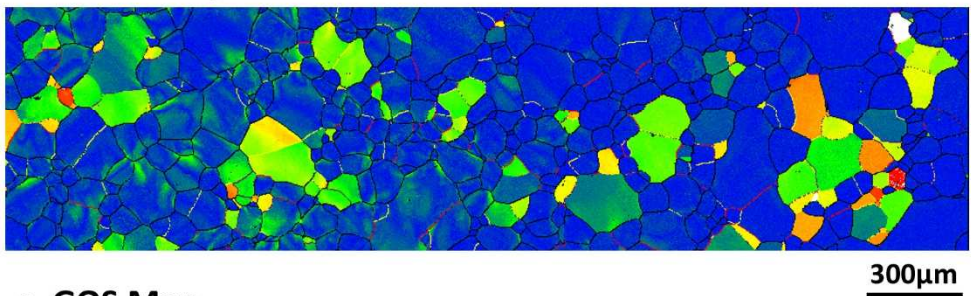


283  
284

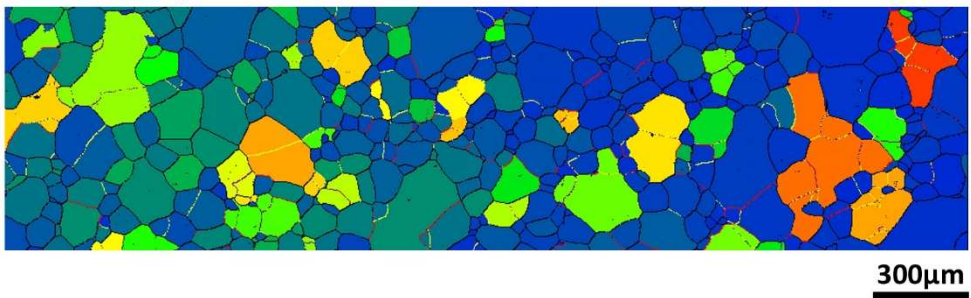
a. IPF Map



b. GROD Map



c. GOS Map



285  
286  
287  
288

Figure 8: IPF in TD direction, GROD and GOS maps of the Zone 2 of the sample

### 3.3. Deeper assessment of the cyclic plastic strain coupling EBSD, ECCI and DIC data

As mentioned previously, the EBSD maps and corresponding measurements give a clear indication of the strain localisation in the deformed notched sample. The LCF behaviour of the non-notched samples has been fully investigated in previous works [32–34] and the obtained results are used to correlate the strain levels measured with DIC and misorientations measured with EBSD for the notched sample to an equivalent total strain variation of non – notched samples.

The main issues from these previous investigations are summarized in figure 9. As the total applied strain increases, the Fe-3% Si steel exhibits behaviours equivalent to low temperature and to high temperature regimes. For the lowest strain amplitudes, dislocation sources are activated within the grain and dislocation slip is planar, as also reported for short range ordered alloys. While restricted by high friction related to the Fe-Si clusters, the dislocation glide length remains sufficient to accommodate the required strain in a quasi-static regime. This leads to transgranular cracking. By contrast, for the highest level of deformation, plastic strains and cumulated strains can successfully reduce the restricted effect of the Fe-Si short range order on the plasticity. Grains accommodate individually plastic deformation, but dislocation glide through grains is difficult. The resulting incompatibility effects produce intergranular stresses and strain localisation that generate intergranular crack initiation (Figure 9a).

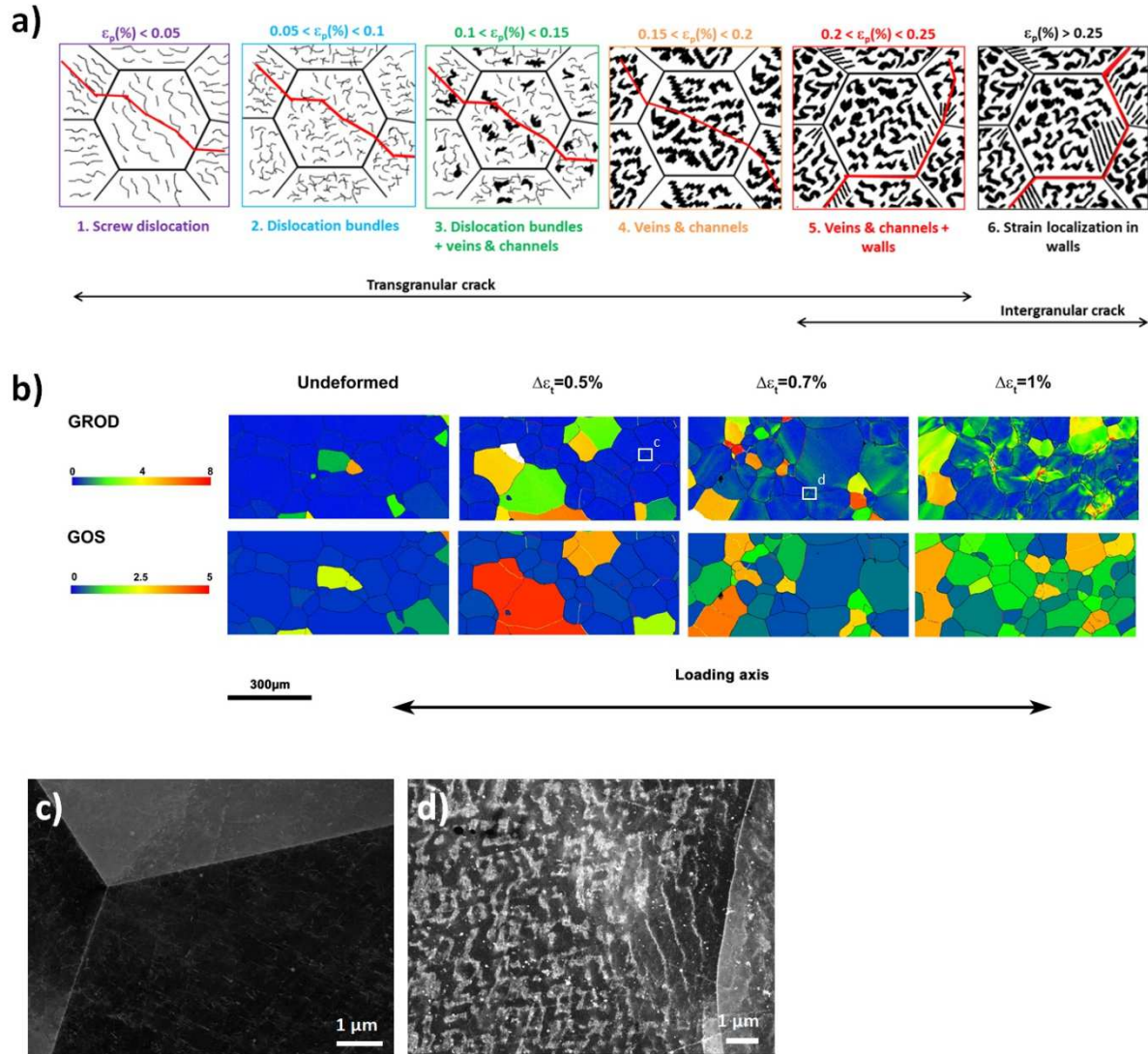


Figure 9: (a) Schematic representation of dislocations structures involved in the damage of the Fe-Si steel (b) Evolution of the GROD and GOS maps for LCF test on smooth specimens as a function of the applied strain (c) and (d) corresponding ECCL observations

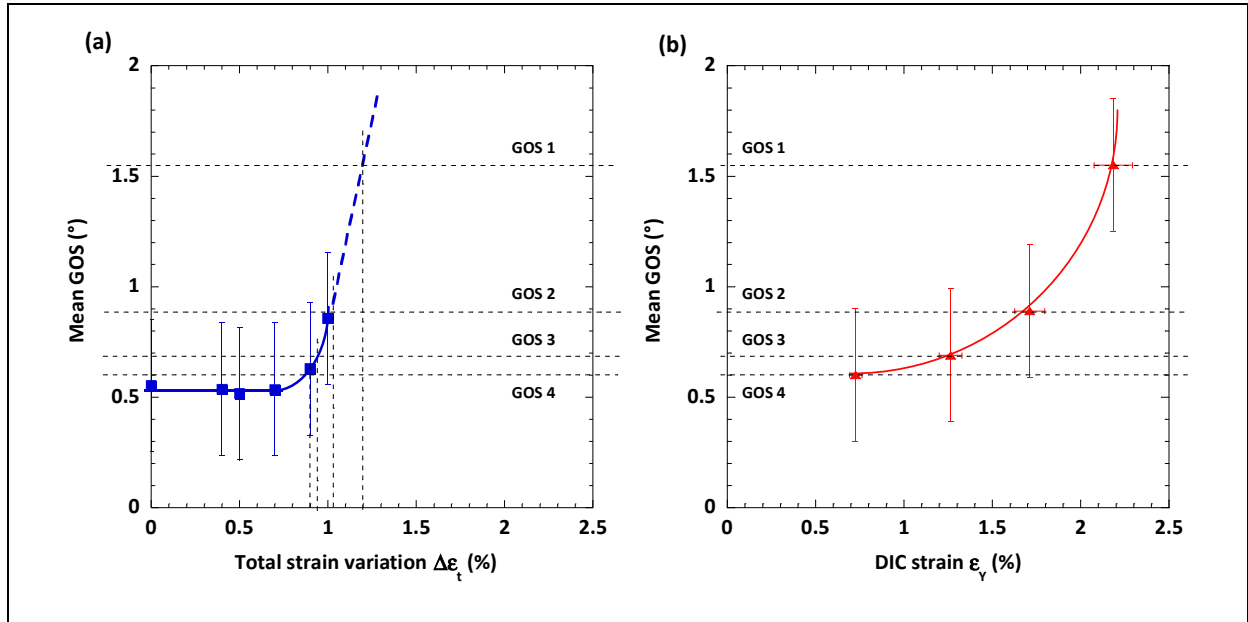
This strain accommodation was suggested from EBSD data processing and imaged by ECCL and two major conclusions were highlighted (Figure 9b, 9c and 9d). Firstly, the strain accommodation with the grains is associated by the formation of dislocation structures commonly described in fatigue [43–45]. Nevertheless, due to the stress incompatibility at the grain boundary, the corresponding strain localisation results in variation of dislocations structure between the grain interiors (vein and channels) and the vicinity of the grain boundary (walls). This difference can be clearly evaluated in

terms of misorientations, since veins consists of an agglomeration of edge dipoles, surrounded by GNDs. By contrast, channels only exhibit very few SSDs. In addition, walls are veins evolutions for which edge dipoles accumulate and then confine while misorientation increases [42,46–48]. Here both step size and angular resolution, considered in the present analysis, do not permit a fine distinction between dislocations rich veins/walls zones and channels. Nevertheless the transition between veins/channels and walls results in gradients that were clearly observed through the GROD criterion [32,49].

On the second hand, a clear correlation between the appearance of a strain localisation as a function of the applied strain level has been evaluated by taking into account the GOS criterion. Indeed, such a strain localisation is correlated to misorientation gradients at the grain interfaces. The deeper the walls structures spread inside the grains, the higher become the gradient. In addition, the number of grains for which such a localisation effect occur also increases with higher total strain levels.

From these observations, there is a way to estimate the equivalent strain undergone by the material in the different areas of zone 1 based on the relation established in figure 10 (a) in which the mean GOS value is correlated to the total strain variation. A numerical correlation is done for an estimation of the GOS value corresponding to  $\Delta\epsilon_t > 1.0\%$ .

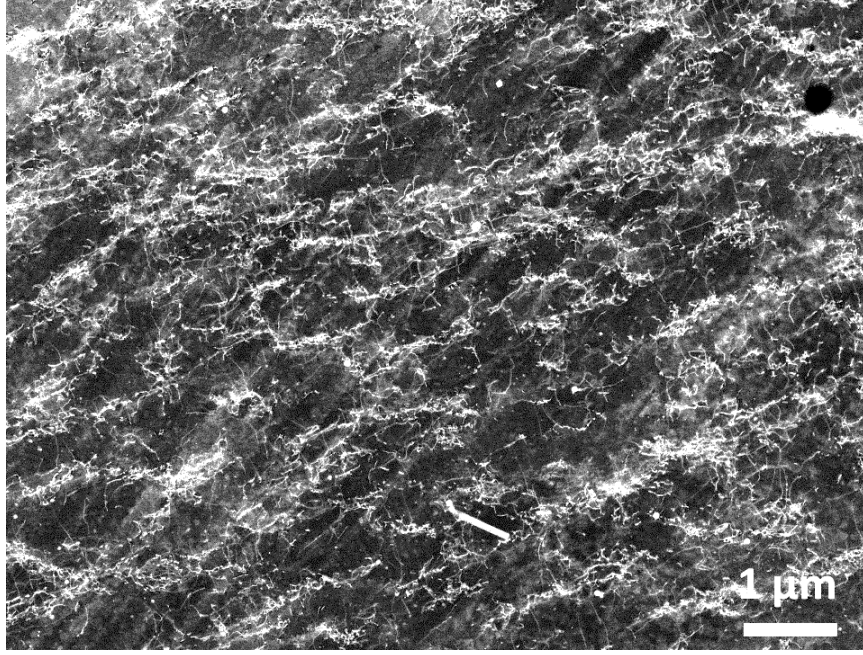
Therefore, the mean GOS values previously measured on the notched specimen in zone 1 are reported in Figure 10 (a) and the equivalent total strain variations are deduced by graphical reading. In addition, these mean GOS values are plotted against the normal local strain value obtained by DIC in Figure 10 (b).



**Figure 10: (a) Equivalent total strain estimation in the different zones of the notched specimen from the relation between GOS and total strain variation established on smooth specimen and (b) mean GOS value of the different zones of the notched specimen versus the normal strain measured by DIC**

It can be noticed here that a direct correspondence of the normal strain calculated by DIC on the notched sample with the total strain variation of the smooth samples is difficult and more especially for the highest strains. Indeed, the LCF study on smooth samples was performed only up to  $\Delta\epsilon_t=1\%$ . Above this strain range, the dislocation structure and mean GOS value in the case of smooth sample are unknown. Also, a precise estimation of the strain by the GOS criteria over a small area is difficult because large grain size hinders statistical analyses and strain measurements by DIC are made on a macroscopic scale.

The observation of the dislocation structure in area 1 of zone 1 (zone where highest strains are reached) by ECCI is shown in Figure 11. It reveals irregular dislocation walls in the process of transforming into dislocation cells.



**Figure 11: ECCL micrograph of the dislocation structure near the notch**

This structure expresses a more advanced deformation state than those observed in smooth samples loaded at  $\Delta\epsilon_t=1\%$ . Therefore, this is consistent with the fact that in area 1, strains superior to the equivalent total strain variation of 1% are reached and that a ratcheting effect, leading to mean strain of 3% is observed.

Furthermore, referring to these new results, one possible evolution of mean GOS as a function of total strain variation is shown in Figure 12. In this figure, the GOS evolution with the total strain variation is depicted in three stages. During the first stage, the mean GOS is constant over a wide strain range (from  $\Delta\epsilon_t=0\%$  to  $\Delta\epsilon_t=0.7\%$ ) because the dislocation structure is homogeneous in the grain. During the second stage, the mean GOS increases rapidly with the total strain variation (from  $\Delta\epsilon_t=0.7\%$  to  $\Delta\epsilon_t=1\%$ ), this is due to localisation of the strain at grain boundary and higher misorientation in this zone.

The linear increase of the mean GOS with  $\Delta\epsilon_t$  reflects the increasing ratio between dipolar walls in the vicinity of grain boundaries and veins in the grain interiors. When the grain is completely filled out with walls, an increase in strain should trigger the transformation of walls into cells. A new regime, the third stage, takes then place.



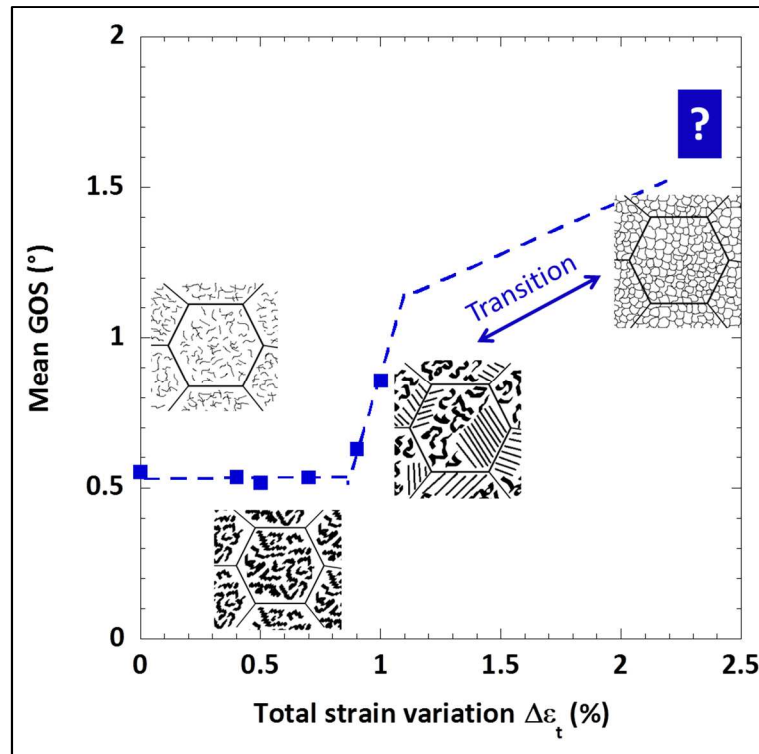


Figure 12: Evolution of mean GOS with total strain variation

The proposed approach, based on a comparison between GROD/GOS misorientation criteria and linked with DIC measurements and ECCI imaging, differs from the current approaches developed around EBSD, ECCI and DIC. Although the selected EBSD acquisition parameters (step size, EBSD resolution) do not permit to detail locally the Nye tensor and calculate precisely the dislocations densities by lacking of both high spatial resolution and high angular resolution, it allows the comparison of different zones in the sample and propose a clear view of the local plasticity levels.

Indeed, the major trends of the new EBSD developments are based on high resolution EBSD (HR-EBSD) or Cross-Correlation EBSD (CC-EBSD). Nevertheless, most of the works do not face directly the calculated GNDs densities, distributions and stress/strain fields with corresponding dislocations structures for FCC structures (single phase copper and nickel alloys) [30,50,51]. A more recent approach makes a comparison between cECCI and CC-EBSD in order to explain how entangled dislocations evolve to wall and channels structure in a DP steel [52]. However, in that latter case, a

huge amount of data, coupled with a fine step size in terms of EBSD acquisition are required. Although the corresponding results are highly valuable and bring microscopic details on how the microstructure evolve during cycling, the scale limitation remains restraint when taking into account a large zone with a possible variation in terms of dislocations structuration as observed in the present study.

The same kind of observation can be done for DIC measurements, as for cyclic plasticity, the measurements are mainly related to the residual strains or the maximum strains, depending on how and when the data are acquired.

## **Conclusions**

The current work aimed at proposing a judicious combination of microscopy related measurements and observations to understand in both qualitative and quantitative ways the effect of cyclic loading on the microstructure response of a Fe-3%Si steel. Three complementary techniques have been considered: EBSD, ECCI and DIC.

Although each of the techniques is well known and its application to plastic strain determination well documented in the literature, some considerations have to be taken into account.

- EBSD analysis, conducting with right selected parameters, can lead to a clear overview of the plastic strain. However, a particular attention has to be done when post-processing the data. In general, EBSD data are only linked to misorientation and pattern quality measurements. The first aspect indirectly characterises the GND dislocations, while the second may be related with both GND and SSD dislocations. The corresponding strain and stress field can also derivate from the misorientation measurements, but the acquisition step-size is of prime importance.



- ECCI gives a clear observation of the dislocations, as these latter would be observed by TEM.

However, this technique cannot make a distinction between SSDs and GNDs.

- Strain fields obtained through DIC measurements are easily understood. When characterising the strain fields coming from a cyclic loading, the situation appears to be a bit more complex.

The combination of the data coming from the three different techniques avoid to make some mistakes. The misorientation criteria obtained with EBSD can easily be associated to dislocations structuration, as comparing with ECCI observations. In a same manner, such measurements can also be related to strain levels determined by DIC.

Applied on a notched specimen made of Fe-3%Si steel, submitted to cyclic loading, new details on the materials behaviour have been obtained.

The DIC measurements clearly emphasise the strain localisation effect during cycling. In addition, it points out a ratcheting effect, which is more pronounced close to the notch. However, taking into account the resulting deformation measurement from the DIC data alone does not reflect the accumulated plasticity of the material. Hence, the use of abacuses, linking local misorientation and total strain variation, on one hand and misorientation and the DIC measurements of residual cyclic strain on the other hand, confirms a higher accumulated cyclic strain in the notch region, which can be related to the combination of a higher strain amplitude with a higher mean strain. This has also been confirmed by the ECCI observations that show irregular dislocation walls in the process of transforming into dislocation cells.

## **Acknowledgment**

The authors thank Valeo France for Financial support, Mr. Golek and Mr. Creton for the technical help. The SEM facility in Lille (France) is supported by the Conseil Régional du Nord-Pas de Calais, and the European Regional Development Fund (ERDF).

431

432

433 **Data availability statement**

434 The raw/processed data required to reproduce these findings cannot be shared at this time due to

435 legal or ethical reasons

436

437

## References

- [1] W. Zhang, Y. Zhang, H. Li, X. Gu, Experimental investigation of fatigue bond behavior between deformed steel bar and concrete, *Cem. Concr. Compos.* 108 (2020) 103515. doi:10.1016/j.cemconcomp.2020.103515.
- [2] M. Tripathi, R.P. Dhakal, F. Dashti, L.M. Massone, Low-cycle fatigue behaviour of reinforcing bars including the effect of inelastic buckling, *Constr. Build. Mater.* 190 (2018) 1226–1235. doi:10.1016/j.conbuildmat.2018.09.192.
- [3] J.-B. Vogt, C. Carle, J. Bouquerel, I. Proriot Serre, Behaviour of short and long cracks in air and in liquid metal in T91 steel, in: *MATEC Web Conf.*, 2018. doi:10.1051/matecconf/201816503016.
- [4] J.B. Vogt, S. Argillier, J.P. Massoud, V. Prunier, Fatigue damage evaluation of a power plant component from analysis of the dislocation structures, *Eng. Fail. Anal.* 7 (2000) 301–310. doi:10.1016/S1350-6307(99)00034-5.
- [5] M. Shirani, G. Härkegård, A review on fatigue design of heavy section EN-GJS-400-18-LT ductile iron wind turbine castings, *Energy Equip. Syst.* 2 (2014) 5–24. doi:10.22059/ees.2014.5008.
- [6] R. Branco, J.D. Costa, F. Berto, A. Kotousov, F. V. Antunes, Fatigue crack initiation behaviour of notched 34CrNiMo6 steel bars under proportional bending-torsion loading, *Int. J. Fatigue.* (2020). doi:10.1016/j.ijfatigue.2019.105268.
- [7] F.M. Firouz, E. Mohamed, A. Lotfy, A. Daoud, M.T. Abou El-Khair, Thermal expansion and fatigue properties of automotive brake rotor made of AlSi–SiC composites, *Mater. Res. Express.* 6 (2020) 1265d2. doi:10.1088/2053-1591/ab6129.
- [8] M. Fonte, P. Duarte, V. Anes, M. Freitas, L. Reis, On the assessment of fatigue life of marine diesel engine crankshafts, *Eng. Fail. Anal.* 56 (2015) 51–57.

doi:10.1016/j.engfailanal.2015.04.014.

[9] Z. Zheng, S. Yuan, T. Sun, S. Pan, Fractographic study of fatigue cracks in a steel car wheel, Eng. Fail. Anal. 47 (2015) 199–207. doi:10.1016/j.engfailanal.2014.09.010.

[10] S.M.O. Tavares, P.M.S.T. de Castro, An overview of fatigue in aircraft structures, Fatigue Fract. Eng. Mater. Struct. 40 (2017) 1510–1529. doi:10.1111/ffe.12631.

[11] L. Li, A. Kedous-Lebouc, A. Foggia, J.C. Mipo, Influence of magnetic materials on claw pole machines behavior, IEEE Trans. Magn. 46 (2010) 574–577. doi:10.1109/TMAG.2009.2032520.

[12] G. Cai, C. Li, B. Cai, Q. Wang, An investigation on the role of texture evolution and ordered phase transition in soft magnetic properties of Fe–6.5 wt%Si electrical steel, J. Magn. Magn. Mater. 430 (2017) 70–77. doi:10.1016/j.jmmm.2017.01.054.

[13] M. Reinlein, T. Hubert, A. Hoffmann, A. Kremser, Optimization of analytical iron loss approaches for electrical machines, 2013 3rd Int. Electr. Drives Prod. Conf. EDPC 2013 - Proc. (2013) 1–7. doi:10.1109/EDPC.2013.6689759.

[14] F. Henrotte, S. Steentjes, K. Hameyer, C. Geuzaine, Iron loss calculation in steel laminations at high frequencies, IEEE Trans. Magn. 50 (2014) 0–3. doi:10.1109/TMAG.2013.2282830.

[15] M. Sugiyama, M. Shibata, Application of Scanning Electron Microscope to Dislocation Imaging in Steel, JEOL News. 46 (2011) 11–16. <http://www.jeol.co.jp/en/applications/detail/780.html>.

[16] S. Zaefferer, N.-N. Elhami, Theory and application of electron channelling contrast imaging under controlled diffraction conditions, Acta Mater. 75 (2014) 20–50. doi:10.1016/j.actamat.2014.04.018.

[17] J. Jiang, T.B. Britton, A.J. Wilkinson, Evolution of dislocation density distributions in copper during tensile deformation, Acta Mater. 61 (2013) 7227–7239. doi:10.1016/j.actamat.2013.08.027.

- 485 [18] J. Jiang, T.B. Britton, A.J. Wilkinson, Measurement of geometrically necessary dislocation  
486 density with high resolution electron backscatter diffraction: Effects of detector binning and  
487 step size, *Ultramicroscopy*. 125 (2013) 1–9. doi:10.1016/j.ultramic.2012.11.003.
- 488 [19] M. Hayakawa, M. Wakita, E. Nakayama, Damage evaluation of ferrite and ferrite-pearlite steel  
489 during fatigue crack initiation by EBSD, *Adv. Mater. Res.* 891–892 (2014) 410–415.  
490 doi:10.4028/www.scientific.net/AMR.891-892.410.
- 491 [20] M.J. Holzweissig, P. Kanagarajah, H.J. Maier, Digital image correlation at high temperatures  
492 for fatigue and phase transformation studies, *J. Strain Anal. Eng. Des.* 49 (2014) 204–211.  
493 doi:10.1177/0309324713498737.
- 494 [21] A.D. Darbal, M. Gemmi, J. Portillo, E. Rauch, S. Nicolopoulos, Nanoscale Automated Phase and  
495 Orientation Mapping in the TEM, *Micros. Today*. 20 (2012) 38–42.  
496 doi:10.1017/S1551929512000818.
- 497 [22] C. Rehrl, S. Kleber, T. Antretter, R. Pippan, A methodology to study crystal plasticity inside a  
498 compression test sample based on image correlation and EBSD, *Mater. Charact.* 62 (2011)  
499 793–800. doi:10.1016/j.matchar.2011.05.009.
- 500 [23] G. L’hôte, C. Lafond, P. Steyer, S. Deschanel, T. Douillard, C. Langlois, S. Cazottes, Rotational-  
501 Electron Channeling Contrast Imaging analysis of dislocation structure in fatigued copper  
502 single crystal, *Scr. Mater.* 162 (2019) 103–107. doi:10.1016/j.scriptamat.2018.10.050.
- 503 [24] M.T. Welsch, M. Henning, M. Marx, H. Vehoff, Measuring the plastic zone size by Orientation  
504 Gradient Mapping (OGM) and Electron Channeling Contrast Imaging (ECCI), *Adv. Eng. Mater.* 9  
505 (2007) 31–37. doi:10.1002/adem.200600195.
- 506 [25] T. Niendorf, J. Dadda, D. Canadinc, H.J. Maier, I. Karaman, Monitoring the fatigue-induced  
507 damage evolution in ultrafine-grained interstitial-free steel utilizing digital image correlation,  
508 *Mater. Sci. Eng. A*. 517 (2009) 225–234. doi:10.1016/j.msea.2009.04.053.

- 509 [26] A. Guery, F. Hild, F. Latourte, S. Roux, Slip activities in polycrystals determined by coupling DIC  
510 measurements with crystal plasticity calculations, *Int. J. Plast.* 81 (2016) 249–266.  
511 doi:10.1016/j.ijplas.2016.01.008.
- 512 [27] W. Abuzaid, H. Sehitoglu, J. Lambros, Plastic strain localization and fatigue micro-crack  
513 formation in Hastelloy X, *Mater. Sci. Eng. A.* 561 (2013) 507–519.  
514 doi:10.1016/j.msea.2012.10.072.
- 515 [28] E. Salvati, S. O'Connor, T. Sui, D. Nowell, A.M. Korsunsky, A study of overload effect on fatigue  
516 crack propagation using EBSD, FIB–DIC and FEM methods, *Eng. Fract. Mech.* 167 (2016) 210–  
517 223. doi:10.1016/j.engfracmech.2016.04.034.
- 518 [29] J. Nellessen, S. Sandlöbes, D. Raabe, Effects of strain amplitude, cycle number and orientation  
519 on low cycle fatigue microstructures in austenitic stainless steel studied by electron  
520 channelling contrast imaging, *Acta Mater.* 87 (2015) 86–99.  
521 doi:10.1016/j.actamat.2014.12.024.
- 522 [30] T. Zhang, J. Jiang, B. Britton, B. Shollock, F. Dunne, Crack nucleation using combined crystal  
523 plasticity modelling, high-resolution digital image correlation and high-resolution electron  
524 backscatter diffraction in a superalloy containing non-metallic inclusions under fatigue, *Proc.*  
525 *R. Soc. A Math. Phys. Eng. Sci.* 472 (2016). doi:10.1098/rspa.2015.0792.
- 526 [31] F. Bridier, J.C. Stinville, N. Vanderesse, P. Villechaise, P. Bocher, Microscopic strain and crystal  
527 rotation measurement within metallurgical grains, *Key Eng. Mater.* 592–593 (2014) 493–496.  
528 doi:10.4028/www.scientific.net/KEM.592-593.493.
- 529 [32] C. Schayes, J. Bouquerel, J.-B. Vogt, F. Palleschi, S. Zaefferer, A comparison of EBSD based  
530 strain indicators for the study of Fe-3Si steel subjected to cyclic loading, *Mater. Charact.* 115  
531 (2016). doi:10.1016/j.matchar.2016.03.020.
- 532 [33] C. Schayes, J.-B. Vogt, J. Bouquerel, F. Palleschi, S. Zaefferer, Cyclic plasticity mechanism of the

533 M330-35A steel, *Int. J. Fatigue*. 82 (2016). doi:10.1016/j.ijfatigue.2015.09.008.

534 [34] C. Schayes, J.-B. Vogt, J. Bouquerel, F. Palleschi, Rotor Design Optimisation through Low Cycle  
535 Fatigue Testing, in: *Procedia Eng.*, 2015. doi:10.1016/j.proeng.2015.12.663.

536 [35] M.F. De Campos, F.J.G. Landgraf, R. Takanohashi, F.C. Chagas, I.G.S. Falleiros, G.C. Fronzaglia,  
537 H. Kahn, Effect of the hot band grain size and intermediate annealing on the deformation and  
538 recrystallization textures in low silicon electrical steels, *ISIJ Int.* 44 (2004) 591–597.  
539 doi:10.2355/isijinternational.44.591.

540 [36] M.F. de Campos, F.J.G. Landgraf, I.G.S. Falleiros, G.C. Fronzaglia, H. Kahn, Texture Evolution  
541 during the Processing of Electrical Steels with 0.5% Si and 1.25% Si, *ISIJ Int.* 44 (2004) 1733–  
542 1737. doi:10.2355/isijinternational.44.1733.

543 [37] M.A. da Cunha, S. da C. Paolinelli, Low core loss non-oriented silicon steels, *J. Magn. Magn.*  
544 *Mater.* 320 (2008) 2485–2489. doi:10.1016/j.jmmm.2008.04.054.

545 [38] A. Ramazani, A. Schwedt, A. Aretz, U. Prah, W. Bleck, Characterization and modelling of  
546 failure initiation in DP steel, *Comput. Mater. Sci.* 75 (2013) 35–44.  
547 doi:10.1016/j.commatsci.2013.04.001.

548 [39] N. McCormick, J. Lord, Digital image correlation, *Mater. Today*. 13 (2010) 52–54.  
549 doi:10.1016/S1369-7021(10)70235-2.

550 [40] Y. Chen, J. Hjelen, S.S. Gireesh, H.J. Roven, Optimization of EBSD parameters for ultra-fast  
551 characterization, *J. Microsc.* 245 (2012) 111–118. doi:10.1111/j.1365-2818.2011.03551.x.

552 [41] S.K. Paul, A critical review of experimental aspects in ratcheting fatigue: Microstructure to  
553 specimen to component, *J. Mater. Res. Technol.* 8 (2019) 4894–4914.  
554 doi:10.1016/j.jmrt.2019.06.014.

555 [42] H. Mughrabi, Dual role of deformation-induced geometrically necessary dislocations with

556 respect to lattice plane misorientations and/or long-range internal stresses, *Acta Mater.* 54  
557 (2006) 3417–3427. doi:10.1016/j.actamat.2006.03.047.

558 [43] F. Ackermann, L.P. Kubin, J. Lepinoux, H. Mughrabi, The dependence of dislocation  
559 microstructure on plastic strain amplitude in cyclically strained copper single crystals, *Acta*  
560 *Metall.* 32 (1984) 715–725. doi:10.1016/0001-6160(84)90145-7.

561 [44] H. Mughrabi, K. Herz, X. Stark, Cyclic deformation and fatigue behaviour of alpha-iron mono-  
562 and polycrystals, *Int. J. Fract.* 17 (1981) 193–220. doi:10.1007/BF00053520.

563 [45] D. Kuhlmann-Wilsdorf, C. Laird, Dislocation behavior in fatigue, *Mater. Sci. Eng.* 27 (1977)  
564 137–156. doi:10.1016/0025-5416(77)90166-5.

565 [46] H. Mughrabi, Deformation-induced long-range internal stresses and lattice plane  
566 misorientations and the role of geometrically necessary dislocations, *Philos. Mag.* 86 (2006)  
567 4037–4054. doi:10.1080/14786430500509054.

568 [47] A. Irastorza-Landa, H. Van Swygenhoven, S. Van Petegem, N. Grilli, A. Bollhalder, S.  
569 Brandstetter, D. Grolimund, Following dislocation patterning during fatigue, *Acta Mater.* 112  
570 (2016) 184–193. doi:10.1016/j.actamat.2016.04.011.

571 [48] A. Irastorza-Landa, N. Grilli, H. Van Swygenhoven, Effect of pre-existing immobile dislocations  
572 on the evolution of geometrically necessary dislocations during fatigue, *Model. Simul. Mater.*  
573 *Sci. Eng.* 25 (2017). doi:10.1088/1361-651X/aa6e24.

574 [49] W. Pantleon, Resolving the geometrically necessary dislocation content by conventional  
575 electron backscattering diffraction, *Scr. Mater.* 58 (2008) 994–997.  
576 doi:10.1016/j.scriptamat.2008.01.050.

577 [50] J. Jiang, J. Yang, T. Zhang, F.P.E. Dunne, T. Ben Britton, On the mechanistic basis of fatigue  
578 crack nucleation in Ni superalloy containing inclusions using high resolution electron  
579 backscatter diffraction, *Acta Mater.* 97 (2015) 367–379. doi:10.1016/j.actamat.2015.06.035.



- 580 [51] J. Jiang, T.B. Britton, A.J. Wilkinson, Evolution of intragranular stresses and dislocation  
581 densities during cyclic deformation of polycrystalline copper, *Acta Mater.* 94 (2015) 193–204.  
582 doi:10.1016/j.actamat.2015.04.031.
- 583 [52] D. An, S. Zaefferer, Formation mechanism of dislocation patterns under low cycle fatigue of a  
584 high-manganese austenitic TRIP steel with dominating planar slip mode, *Int. J. Plast.* 121  
585 (2019) 244–260. doi:10.1016/j.ijplas.2019.06.009.
- 586