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1            Toward an automated tool for dislocation density  
2            characterization in a scanning electron microscope

3  
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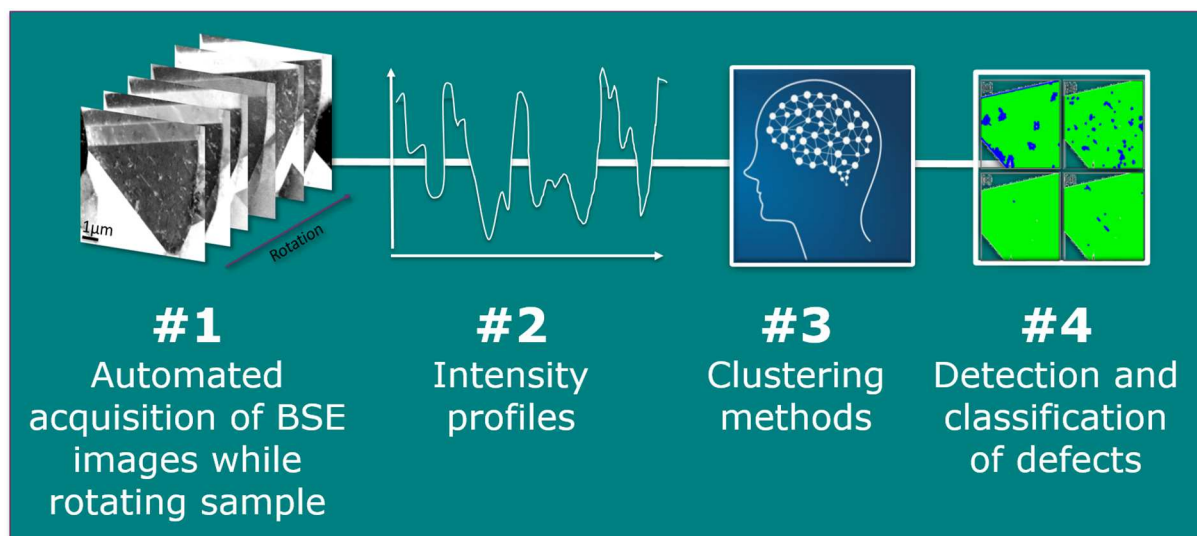
6  
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10

11    Graphical abstract



12

13

14    **Keywords:** Electron channeling contrast imaging; dislocations; scanning electron microscopy;  
15    data clustering

16

17

## 1 **Abstract**

2           We propose a methodology for quantitative dislocation characterization of a bulk  
3 sample in a scanning electron microscope without requiring pre-orientation of the sample  
4 before analysis. In this method, a series of backscattered electron images are acquired while  
5 rotating the sample, and an intensity profile as a function of the rotation angle is obtained for  
6 each pixel of the observed area. These intensity profiles are used to determine the  
7 orientation condition of the analyzed grain. The nature of the pixel is defined as what  
8 dominates the pixel intensity (matrix, defect or noise). As the intensity profiles are also  
9 characteristic of the pixel nature, a data clustering algorithm is applied to the intensity  
10 profiles to classify the pixel nature. As a result, the defect density, such as the dislocation  
11 density, can be automatically measured. The proposed method is fast and efficient compared  
12 with transmission electron microscopy analysis and could enable the future characterization  
13 of multiple grains in a deformed sample within a reasonable amount of time.

14

## 15 **Introduction**

16           During the past decade, considerable efforts have been devoted to the development  
17 of integrated modeling approaches covering material processing to the final material  
18 properties [1]. In these approaches, the microstructural parameters (*e.g.*, precipitate  
19 distribution, grain size) are calculated for a given thermo-mechanical process and used as  
20 input for modeling of the mechanical properties. However, a key parameter required for  
21 predicting the mechanical properties, the dislocation density, remains improperly and rarely  
22 predicted. There is thus a crucial need for accurate experimental measurement of the  
23 dislocation density.

24           Upon all methods available, dislocation density can be measured by transmission  
25 electron microscopy (TEM), electron channeling contrast imaging (ECCI), or by electron back  
26 scattered diffraction (EBSD). TEM allows a direct observation of dislocations with a very good  
27 spatial resolution. Therefore, individual dislocation can be relatively easily well resolved, so  
28 that relatively high dislocation densities can be characterized ( $\sim 10^{15} \text{ m}^{-2}$ ) [2]. However, the  
29 observed area in TEM is quite limited, and in the case of low dislocation densities or  
30 heterogeneous microstructure, those measurement become time consuming. On the other

1 end, the development over the last two decades in EBSD detector sensibility and indexing  
2 speed allowed a fast development of the use of EBSD for the determination of geometrically  
3 necessary dislocation (GND) [3]. This method is particularly useful for high dislocation  
4 densities [4]. Its precision is however limited by the angular resolution and related to the step  
5 size used. Recently, the efficiency of electron channeling contrast imaging (ECCI) has been  
6 demonstrated for defect characterization of bulk samples in a scanning electron microscope  
7 using the channeling contrast of electrons [5-11]. It is now possible to characterize dislocation  
8 densities [5], the nature of dislocations [7], and the nature of stacking faults (SFs) for different  
9 types of materials. ECCI is particularly well adapted for heterogeneous microstructures as it  
10 allows large field of views. It is also widely used to characterize dislocation structure in  
11 fatigued materials [12-14] or specific dislocation/ microstructure interactions [15-16]. ECCI is  
12 particularly suitable for characterizing low dislocation densities. Indeed, for large dislocation  
13 density, dislocations will overlap, which makes the measurement impossible. Most of the  
14 studies on metallic materials present samples with dislocation densities lower than  $1-5 \times 10^{14}$   
15  $m^{-2}$ .

16 There are several available methods to measure the dislocation density of a grain  
17 [11,17,18]. All of these methods are based on counting the dislocations visible in the area of  
18 interest. The counting is often performed manually on individual images. The TEM and ECCI  
19 defect study is based on the  $\vec{g} \cdot \vec{b}$  method and the analysis of invisibility criteria [19] ( $\vec{b}$  is the  
20 Burgers vector of the dislocation and  $\vec{g}$  is the diffracting vector). Indeed, when the matrix is in  
21 a so-called "Two-Beam" condition, a single diffraction vector  $\vec{g}$  is excited. If a small positive  
22 deviation from this orientation is introduced, the dislocation contrast becomes enhanced, the  
23 matrix appears dark, while dislocations appear white. In the case of a screw dislocation, if the  
24 product  $\vec{g} \cdot \vec{b} = 0$  the dislocation is invisible and if the product  $\vec{g} \cdot \vec{b} \neq 0$  then the dislocation is  
25 visible. In order to determine the Burgers vector of a screw dislocation, it is therefore  
26 necessary to observe it under different two – beam conditions. With at least two invisibility  
27 conditions, it is possible to determine its Burgers vector. The approach is similar for edge  
28 dislocations, with the following invisibility condition:  $\vec{g} \cdot \vec{b} \neq 0$  and  $\vec{g} \cdot \vec{b} \wedge \vec{u} \neq 0$ . In the case of  
29 stacking faults, the extinction condition becomes  $\vec{g} \cdot \vec{R} \neq 0$  with  $\vec{R}$  the translation vector  
30 induced by the presence of the defect. Note that in the case of ECCI observations, stress

1 relaxation occur on dislocations that and can modify the visibility/invisibility conditions for  
2 mixed dislocations [20].

3         Reliable characterization of the dislocation density and Burgers vector thus requires  
4 several measurements under different two beam conditions. As diffraction conditions are  
5 specific for each grain, this orientation procedure must be repeated for each analyzed grain.  
6 Consequently, TEM and/or ECCI observations are time consuming, and often, only a few  
7 grains are analyzed. The representativeness of such measurements is thus questionable.  
8 Furthermore, it is well known that the deformation state is orientation dependent, and a  
9 minimum number of grains with different orientation must be characterized.

10         This paper presents an alternative method for quantitative dislocation imaging and  
11 characterization based on the CHanneling ORientation Determination (CHORD) set-up that  
12 does not require any preliminar specific orientation of the sample. The CHORD technique  
13 utilizes a set of ion-induced secondary electron images (iCHORD)[21] or ECCI images  
14 (eCHORD) [22] to automatically map the crystallographic orientation of grains. For eCHORD,  
15 subsequently referred to CHORD, the sample is tilted with respect to the electron beam and  
16 then rotated 360°, with an image recorded for every rotation step. After alignment of the  
17 series of images, an intensity profile as a function of the rotation angle is obtained for each  
18 pixel of the area of interest. This intensity profile is an unequivocal signature of the  
19 orientation of the grain. Then, the crystallographic orientation of each pixel is deduced by  
20 comparison of this profile with a simulated electron channeling pattern (ECP). Indeed, an ECP  
21 represents the BSE intensity collected for different angles between the crystal and the  
22 electron beam [23-24]. The ECP are simulated from EMsoft simulation suite from M. De  
23 Graef *et al.* [25][26]. In our characterization approach, the CHORD procedure is used to  
24 identify the crystallographic orientation of the grain and to determine the orientation  
25 condition leading to dislocation observation. When the electron beam is located at the edge  
26 of a Kikuchi band, a two beam condition is reached [11], and the Bragg condition is fulfilled  
27 [24]. If a slight deviation from this Bragg orientation is applied, defects contrast can be  
28 maximized. For instance, if a positive deviation is applied, the matrix appears dark and  
29 dislocations appear with a bright contrast, which corresponds to a minimum in the intensity  
30 profile. The rotational-ECCI (R-ECCI) method involves the rapid acquisition of a 360° rotation  
31 image to obtain the intensity profile of the grain under study [13]. From the intensity profile,

1 the rotation angles corresponding to intensity minima are determined. The sample is then  
2 rotated to reach one of these two beam conditions and the dislocation structure can be  
3 imaged. The dislocation density can then be determined from such images. However, as only  
4 one two beam condition is considered, in which some dislocations may be in the extinction  
5 condition, the density measurement may be biased. That's why our procedure take into  
6 account several two beam conditions, leading to a more exhaustive approach of dislocation  
7 types.

8           Dislocation fields are generally determined using TEM characterization, which requires  
9 tedious sample preparation that can affect the dislocation structure and focuses on very small  
10 areas. Using scanning electron microscopy (SEM), the observed area is more representative of  
11 the material; that's why we propose to automatically characterize and quantify defects by  
12 exploiting CHORD intensity profiles. Indeed, the intensity of defect and matrix pixels differs  
13 when the matrix is in the two beam condition. Direct determination of the pixel nature can  
14 thus eliminate the manual image processing step necessary for measurement of the  
15 dislocation density and characterization of the dislocation nature.

16           The aim of this paper is to present the possibilities and limits of using the intensity  
17 profiles, rather than images, to characterize dislocations in a scanning electron microscope.  
18 The complete acquisition procedure and the use of advanced clustering methods for the data  
19 treatment are presented. The results obtained for a duplex stainless steel are presented as an  
20 example, and the future potential of this procedure is discussed.

21

## 22 **Experimental set-up**

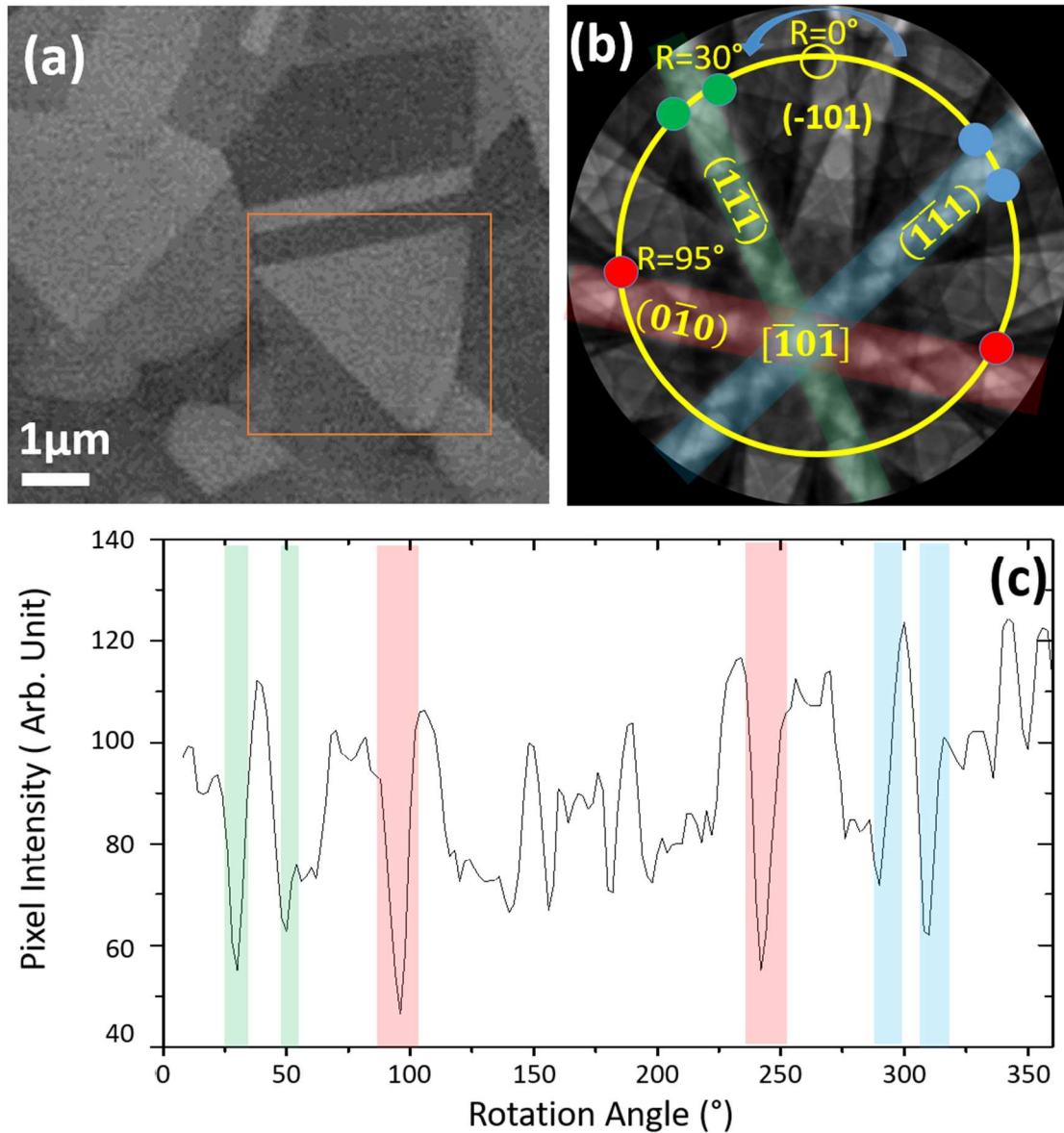
23           The experimental set-up used in this work is described in [22]. The specimens were  
24 mounted to a dedicated sub-stage placed in a classical scanning electron microscope  
25 goniometer to enable fast and precise control of the displacement and rotation (on the order  
26 of nanometers and microdegrees, respectively). The sub-stage was controlled using software  
27 developed by our group to enable automatic rotation–acquisition. Backscattered electron  
28 (BSE) images were automatically acquired for every rotation step of the sample using a  
29 standard bottom column BSE detector. A scan rotation correction was performed at each

1 acquisition to ensure that the exact same region of interest was examined for each  
2 acquisition step.

3         The observation conditions were those typically used for ECCI (Zaefferer and Elhami,  
4 2014). Serial imaging was performed using a Gemini I SEM column (Carl Zeiss Microscopy  
5 GmbH, Oberkochen, Germany) equipped with a retractable standard four-quadrant solid-  
6 state detector (4Q-BSD). The accelerating voltage was set to 20 kV, the aperture size was 120  
7  $\mu\text{m}$ , and the so-called high-current mode was activated. A tilt angle value of  $10^\circ$  and a WD of  
8 7mm were chosen, as in Lafond *and al* work [22]. For this set-up, the beam current is  
9 estimated to be around 10nA [11]. Two images series were recorded with an image size of  
10 1024 x 768 pixel: the first one, at low magnification (pixel size = 22.3 nm ), and with a fast  
11 scan speed ( dwell time = 1.74 ms – 1.4sec/frame), and the second one at higher  
12 magnification ( pixel size = 5.6 nm) and a slower scan speed (dwell time = 26.32ms –  
13 20sec/frame).

14         The sample studied was as-received commercial 2101 lean duplex stainless steel from  
15 Outokumpo; the microstructure of the sample is shown elsewhere [27]. The sample was  
16 prepared by mechanical polishing down to 1  $\mu\text{m}$  followed by a vibratory polishing step using a  
17 colloidal-type suspension. This material was selected because of its small grain size (5–10  
18  $\mu\text{m}$ ). As the sample is undeformed, its dislocation density is expected to be quite small,  
19 around  $10^{12}$  to  $10^{13} \text{ m}^{-2}$ .

20



1

2 *Figure 1: Fast CHORD image series recorded for the ROI; rotation step = 2°, tilt = 10°, pixel size*  
 3 *- 22.3 nm, and accelerating voltage = 20 kV. (a) One of the collected BSE images, (b) calculated*  
 4 *ECP of the selected grain using EMSOFT [25][26], and (c) corresponding experimental intensity*  
 5 *profile. At a sample rotation angle of 30° and 50°, the  $(1\bar{1}\bar{1})$  band is crossed, while the  $(0\bar{1}0)$*   
 6 *band is crossed for  $R=95^\circ$  and  $R=245^\circ$ . The  $(\bar{1}\bar{1}1)$  band is crossed for  $R=390^\circ$  and  $R=315^\circ$ .*

7

8 The first step of the procedure consists of acquiring a fast CHORD image series of the  
 9 region of interest (ROI). After image alignment, an intensity profile as a function of the

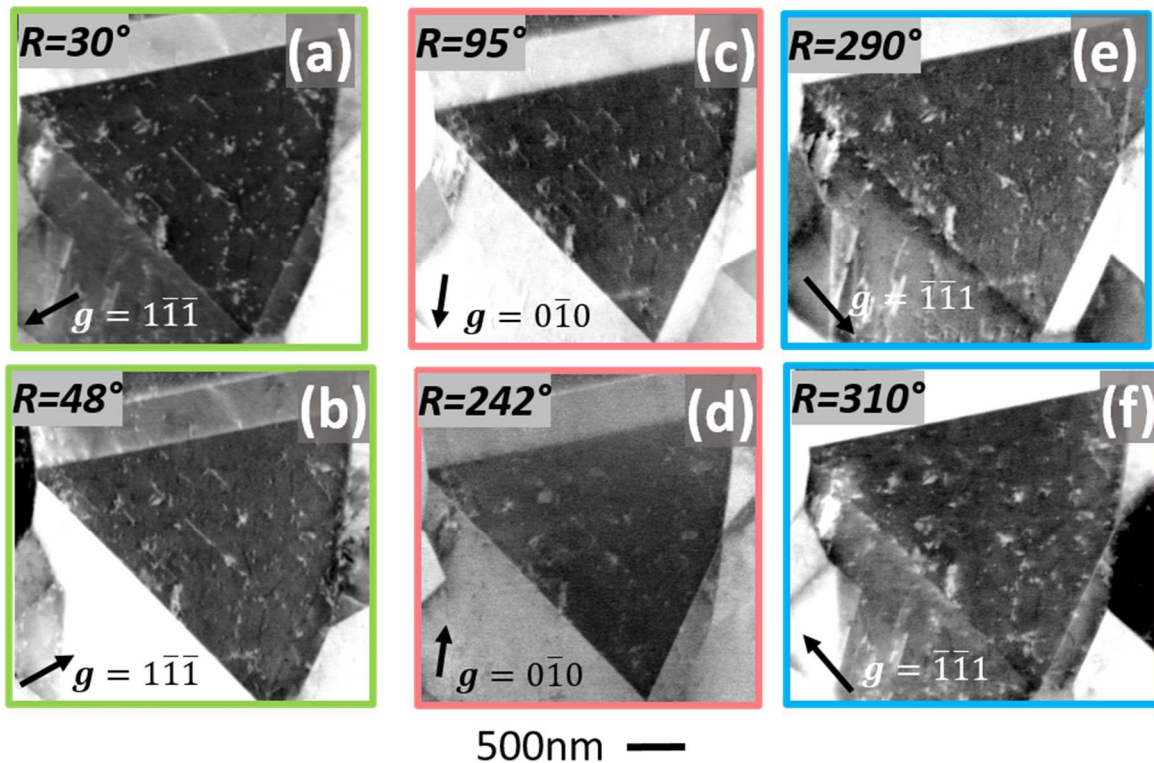


1 rotation angle is available for each pixel of the ROI. From this profile, the grain orientation is  
2 deduced (see [22] for additional details).

3 Figure 1a presents a BSE image of a ROI as an example; the white triangular grain was  
4 selected for dislocation imaging. The rotation series of this ROI was acquired in less than 5  
5 min using the protocol described in [22]. The intensity profile of the whole selected grain is  
6 shown in Figure 1c. The intensity profile was correlated to the ECP presented in Figure 1b,  
7 where the yellow circle represents the electron beam path followed during the 360° sample  
8 rotation with a tilt angle of 10°. It is apparent that this path crosses different Kikuchi bands  
9 such as the  $(1\bar{1}\bar{1})$  band at a rotation angle of about 30° and the  $(0\bar{1}0)$  band at a rotation  
10 angle of about 95°. The intensity profile present a minimum close to those particular rotation  
11 angles, which corresponds to the optimum orientation for dislocation imaging, where the  
12 matrix will appear dark and dislocations bright. Therefore, to visualize dislocations, the  
13 rotation angles needed to reach those maximum contrasts orientation can be directly read on  
14 the intensity profile at the rotation angles corresponding to minimum values. This process can  
15 be used to rapidly find suitable conditions under which some dislocations are visible, without  
16 requiring any electron backscatter diffraction analysis or pre-orientation of the sample. This  
17 approach is thus rapid and allows a first imaging of the dislocation structure [13].

18 However, to quantitatively measure the dislocation density or to determine the  
19 Burgers vector of the present dislocations, imaging under more than one two beam condition  
20 is necessary. By acquiring images for all the rotation angles, *i.e.*, 360 images, many two beam  
21 conditions are assumed to be reached, which should allow quantitative dislocation density  
22 measurement as well as **g.b** analysis. However, practically, a low scan speed (30 s to 2 min  
23 acquisition per frame) is required to visualize the dislocations in the ECCI images of the series.  
24 The total acquisition time is thus very long and may lead to sample drift and loss of focus over  
25 time. Moreover, because of the high magnification required (pixel size < 5–10 nm), a fully  
26 automatic procedure is not yet possible as scanning instabilities causes images distortion.  
27 Therefore, a semi-automatic procedure was preferred: the interesting two beam conditions  
28 were determined from the intensity profile (six conditions for that particular grain), and mini-  
29 rotation series were acquired around those conditions. For each mini-rotation series, a  
30 rotation step of 0.5° was used with the total rotation angle varying from 5° to 10° (see Figure  
31 2). The acquisition speed for all the images was 30 s/image, which is quite fast for ECCI, and

1 the magnification was 20kX (pixel size = 5.6 nm). The observed dislocation width was 20-25  
2 nm, which corresponds to 4 or 5 pixels.



3  
4 *Figure 2: (a)–(f) Sum images from the mini-series (over four images centered on the best*  
5 *diffraction condition – total rotation angle of 2°) for the six two beam conditions selected.*

6

7

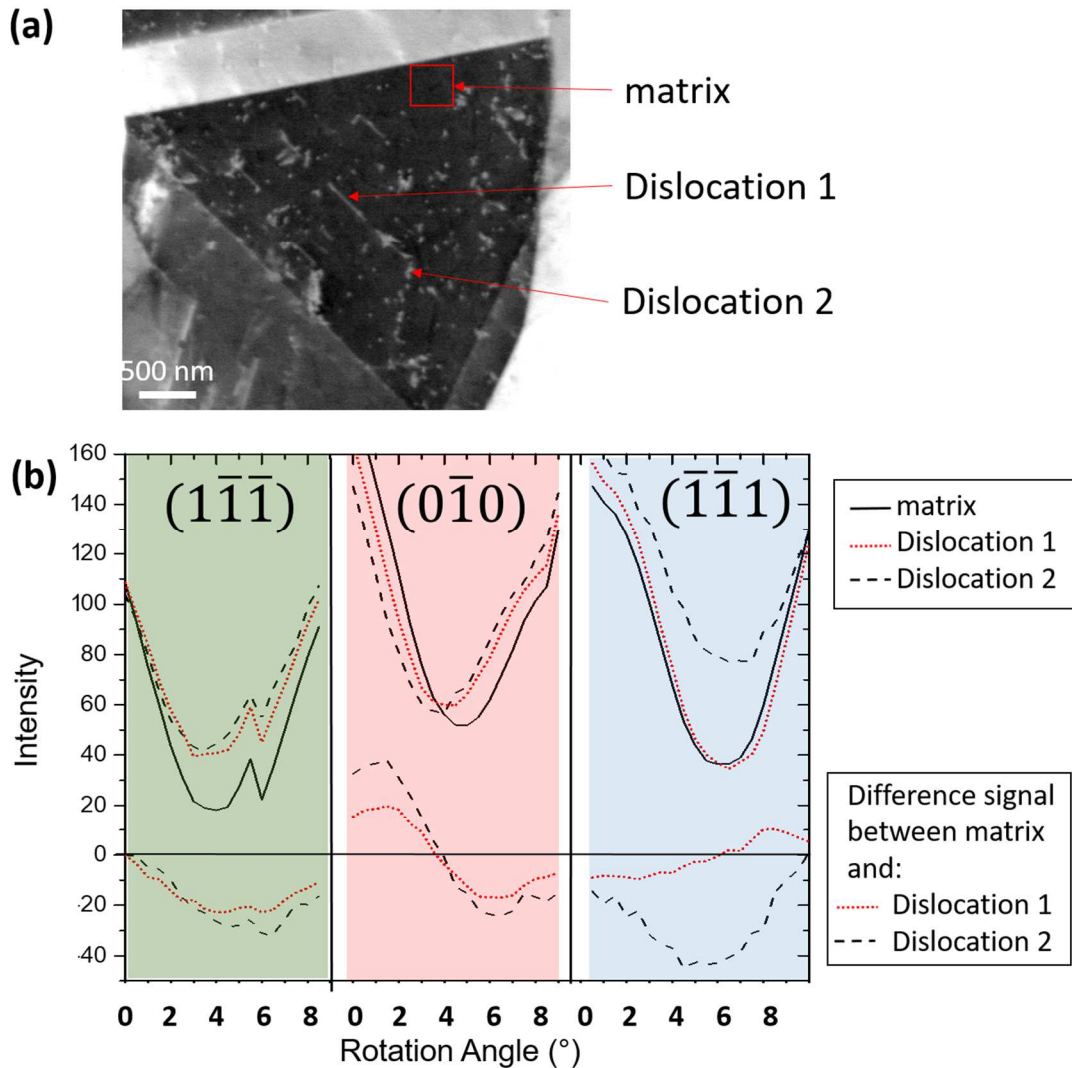
## 8 Results

### 9 1. Intensity profiles as defect markers

10 Figure 2 shows the dislocation structure for all the selected rotation angles. Most dislocations  
11 were visible for the  $g = 1\bar{1}\bar{1}$  condition, with some dislocations disappearing for the  $g = \bar{1}\bar{1}\bar{1}$   
12 and  $g = 0\bar{1}0$  conditions.

13 The use of the intensity profiles to automatically detect the different defects instead of  
14 independent ECCI images was then considered. All the mini-series images were gathered to  
15 create a new stack of images. Then, the stack was aligned in Fiji software using affine  
16 transformations. The complete alignment procedure is detailed in [22]. Thanks to the use of a

1 rotation correction during image acquisition, a precise enough alignment can be achieved,  
 2 considering the pixel size ( 5.8nm). Therefore, an intensity profile as a function of the image  
 3 number was generated for each pixel. As an example, Figure 3 presents parts of the complete  
 4 intensity profile corresponding to the  $g = 1\bar{1}\bar{1}$ ,  $g = 0\bar{1}0$ , and  $g = \bar{1}\bar{1}\bar{1}$  conditions for the  
 5 matrix and for two selected dislocations.



6  
 7 *Figure 3: (a) Selection of the matrix pixels (red square) and of pixels of two different*  
 8 *dislocations to generate corresponding intensity profiles (b). The difference signal between the*  
 9 *matrix and defects are also presented at the bottom.*

10 The intensity profiles of the matrix and of the dislocations close to the three two beam  
 11 conditions were similar to those calculated in [11]. The signal differences between the matrix  
 12 and the selected dislocations are also shown in Figure 3. The matrix pixels are the one located

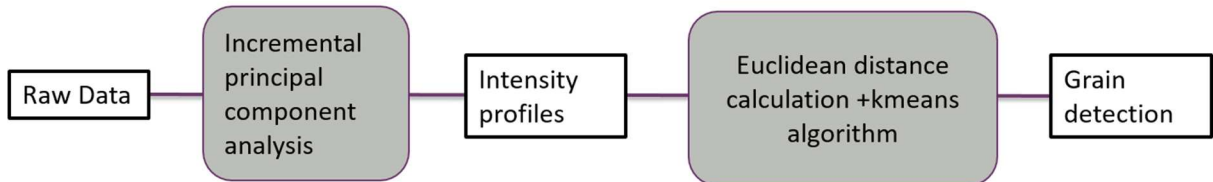
1 inside the red square, while a couple of pixels located at the position of the red arrays were  
2 selected for the two dislocations. The intensity difference of the dislocation signal was  
3 approximately 20%–40% of the matrix intensity, which is more than sufficient to efficiently  
4 discriminate pixels attributed to the matrix from those attributed to dislocations. It is worth  
5 noting that the maximum intensity difference appeared at the minimum contrast condition of  
6 the matrix, as expected from ECCI theory [11], as observed for the  $\mathbf{g} = \bar{1}\bar{1}1$  condition. This is  
7 however not so clear for the  $\mathbf{g} = 1\bar{1}\bar{1}$  condition where the signal difference is maximum and  
8 constant over 2 degrees around the minimum contrast condition. This result indicates that the  
9 dislocations are visible for a relatively large angular spread (more than 5° in this case), which  
10 could result from the crystallographic path followed during the rotation. The beam crosses  
11 the  $\mathbf{g} = (\bar{1}\bar{1}1)$  plane almost perpendicularly, whereas the  $\mathbf{g} = (1\bar{1}\bar{1})$  plane is crossed with  
12 an incident angle close to 10°. Therefore, the minimum contrast condition for the  $\mathbf{g} =$   
13  $(1\bar{1}\bar{1})$  plane and the difference signal are more angularly spread as a function of the rotation  
14 angle. For the  $\mathbf{g} = (\bar{1}\bar{1}1)$  condition, the intensity profile of the dislocation 1 is well  
15 superimposed with the one of the matrix. This finding is consistent with the fact that  
16 dislocation 1 was not visible in the images for that diffracting condition. Dislocation 2 signal  
17 present a similar profile as the matrix, but the intensity of its minimum is different: dislocation  
18 2 is visible in that diffraction condition. For the  $\mathbf{g} = (0\bar{1}0)$  diffracting condition, the intensity  
19 signal of dislocation 1 and of dislocation 2 are almost superimposed with that of the matrix,  
20 even if a slight shift in the peak is observed. This similarity resulted in a difference signal with  
21 an intensity of less than 5% and a flat shape, indicating that the dislocations are extinguished  
22 for this rotation angle, although a slight contrast is visible in the image. When a dislocation  
23 appears in a TEM or ECCI image, a residual contrast is often observed even if the defect is in a  
24 so-called extinction condition, which can lead to an incorrect interpretation of the extinction  
25 condition. The signal difference shape of the intensity profile provides a quantitative tool to  
26 distinguish between extinction or visibility conditions. This example proves that intensity  
27 profiles can be of great assistance in identifying matrix and dislocation pixels and in classifying  
28 defects by their nature, as explained in the following section.

## 29 2. Clustering method for defect detection

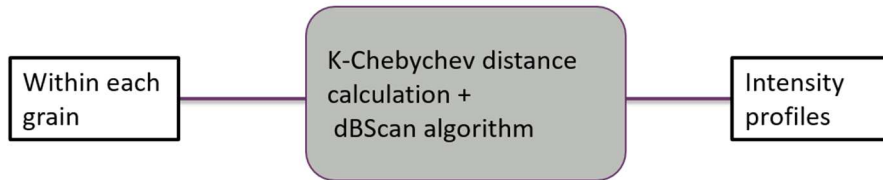
1 The proposed clustering method for defect detection consists of two main steps: first, the  
2 different grains are separated, and then, the defects in each grain are detected and  
3 characterized. The process is summarized in Figure 4.

4

### (1) Segmentation of grains



### (1) Defect detection within a grain



5

6

Figure 4: Image analysis process.

7

## 8 2.1 Segmentation of grains

9 The initial data are represented by the set of intensity profiles for every pixel in the  
10 images. The profile associated with a (X,Y) coordinate is a N-dimensional vector, where N is  
11 the number of images. The clustering method is based on the calculation of the distance in  
12 the N-space between all intensity profiles.

13 A data set contains up to 360 images, each of which contains up to 3 million pixels.  
14 Therefore, running the clustering over the entire data set might require the processing of  
15 more than 4GB of data. However, some information is highly redundant in the profiles over a  
16 grain. Thus, to reduce the size of the data requiring processing, a principal component  
17 analysis (PCA) is used. A PCA projects the data onto a lowered dimensional space. In this case,  
18 an approximation of the PCA, the incremental PCA, was preferred [28]. It allows the principal  
19 component to be built incrementally using batches of a given size and reduces the memory  
20 needed to build the PCA. Once the size of the data was reduced, clustering was performed

1 using a k-means algorithm that makes use of a kd-tree representation of the data points and  
2 the Euclidean distance as a metric [29].

3 Once the cluster was calculated, post-processing operations were performed to merge or split  
4 undesirable clusters. As a result, the grain analysis was efficient when the number of grains  
5 was small, which is suitable for the high magnifications required for dislocation imaging.

6

## 7 2.2 Defect detection within a grain

8 The next step of the procedure consists of labeling each profile within a selected grain.  
9 Three labels were selected: defect, matrix, and noise. Matrix pixels correspond to all the  
10 positions (X,Y) in the analyzed stack that shared similar profiles along all dimensions. Defect  
11 profiles present dissimilarities compared with the average matrix profile only on a series of  
12 consecutive dimensions. Noise profiles are either flat profiles or profiles that do not fit into  
13 the other two categories. The number of defects present within a grain is undetermined  
14 before the clustering process; therefore, a non-knowledge-based algorithm is required.

15 The following clustering procedure was applied: first, a similarity score between  
16 profiles,  $d_{kC}(p, p')$ , was calculated. Provided a span parameter  $k$ , this score is given by the  
17 maximal cumulative difference in the intensity spread over  $k$  consecutive degrees. A defect  
18 differs the most from the matrix on a window of consecutive dimensions; therefore, it is more  
19 appropriate to use a variant of the Chebychev distance, where averages of the coordinates  
20 over consecutive dimensions are used rather than the direct use of the coordinates.

$$22 \quad d_{kC}(p, p') = \max_{1 \leq i \leq n-k} \left| \frac{1}{k} \sum_{j=i}^{i+k-1} p_j - \frac{1}{k} \sum_{j=i}^{i+k-1} p'_j \right|$$

21 (1)

23 Here,  $k$  is the window size;  $d_{kC}$  is the  $k$ -window Chebychev distance, where  $k=1$  is the usual  
24 Chebychev distance;  $p$  and  $p'$  are two different profiles; and  $n$  is the dimension of the  
25 intensity profile, which coincides with the number of images considered.

26 Note that we can replace each profile  $p$  by a profile

$$x_k = \left( \frac{1}{k} \sum_{j=0}^{k-1} p_j, \dots, \frac{1}{k} \sum_{j=n-k}^{n-1} p_j \right).$$

1 (2)

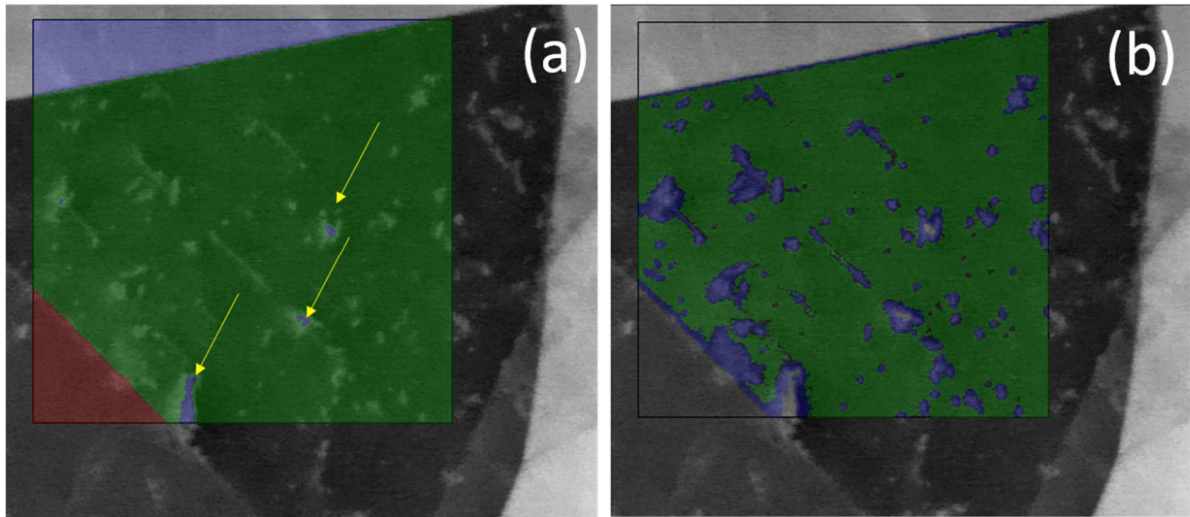
3 This means that the k-window Chebychev distance between two profiles ( $p, p'$ ),  $d_{kC}(p, p')$ ,  
 4 is equivalent to the Chebychev distance  $d_C(p_k, p'_k)$ . A kD-tree representation can thus be  
 5 used together with a clustering algorithm optimized for the Chebychev distance. This is the  
 6 case for the *dbscan* cluster algorithm [30], which is a non-knowledge-based and fast  
 7 algorithm. After labeling, the largest cluster is then considered to be the matrix, and the  
 8 defects are classified depending on the angle at which the difference is calculated. Defects  
 9 are thus classified from their visibility conditions.

10 As the clustering is performed on the profiles and not using the (X,Y) coordinates, it is possible  
 11 for several features to be engulfed in the same cluster. For post-processing, to separate  
 12 defect clusters that are not adjacent in the images, the connected components of the (X,Y)  
 13 positions were computed for each cluster of profiles that was not labeled matrix. Small,  
 14 connected components were discarded as noise, and for the rest, each profile was compared  
 15 with the matrix average profile. If the difference was large enough, the profile was set as part  
 16 of the feature; otherwise, it was considered as noise. The difference value was set as a  
 17 variable parameter, adjusted by the user.

18

### 19 **3. Application on a polycrystalline duplex steel: dislocation density measurements**

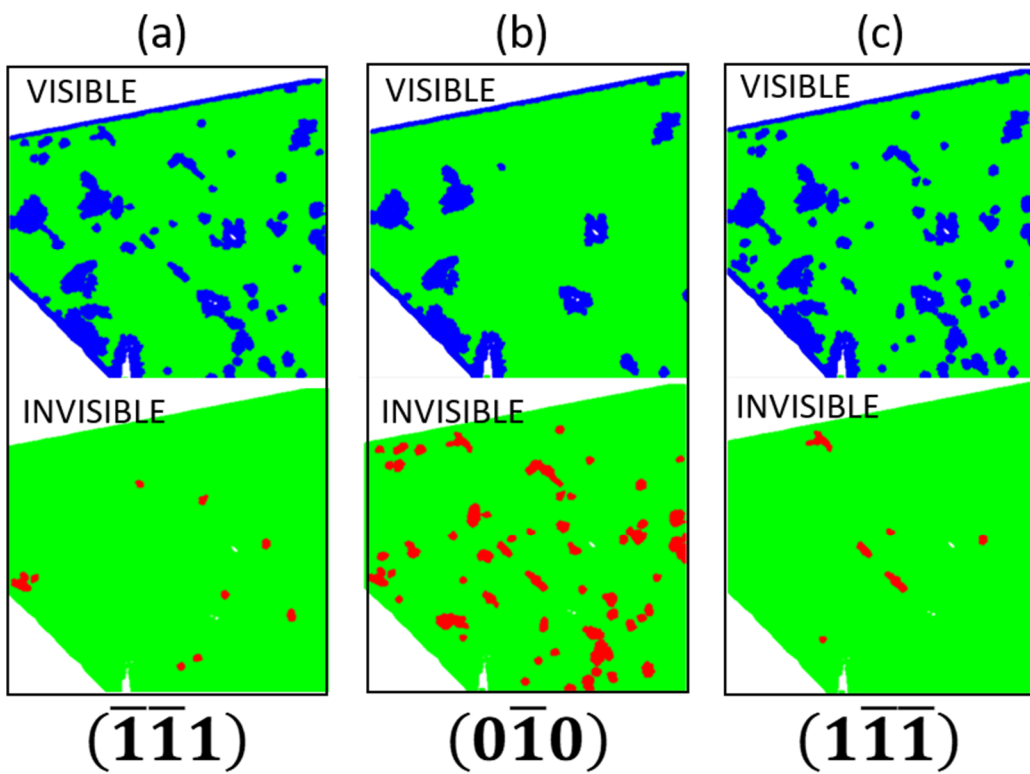
20 The mini-series of images were analyzed using the clustering method described above. In  
 21 the ROI, three different grains were detected, corresponding to three clusters (Figure 5a).  
 22 Note that some features, indicated by the yellow arrows in Figure 5, were not included in the  
 23 green grain. These features exhibited high contrast and were visible in all the images, even far  
 24 away angularly from the Bragg condition. Recording a 360° image series would be necessary  
 25 to determine if those features are topographical defects or dislocation type defects. Then,  
 26 within each grain of interest, defect detection was performed (Figure 5b). The pixels were  
 27 classified by type (matrix, defect, or noise), and the angles for which the contrast between the  
 28 matrix and defect differed were determined. These angles correspond to the visibility  
 29 conditions.



1

2 *Figure 5: (a) Clustering of the mini-series used to detect the different grains (clusters) and (b)*  
 3 *defect detection within the selected cluster.*

4 4. Determining the defect nature



5

6 *Figure 6: Visible (blue) and invisible (red) defects for the three diffracting conditions.*

7



1 All the defects were classified from their visibility/invisibility criterion. In the example of  
 2 Figure 6, the visible defects are shown in blue and the invisible defects are shown in red for  
 3 the three diffracting conditions. This type of representation enables a direct comparison of  
 4 the feature detection and original images. For this grain, the highest number of defects are  
 5 visible for  $\mathbf{g} = \bar{1}\bar{1}1$ , in the image series as well as after profile analysis. Moreover, many  
 6 dislocations are not seen in the  $\mathbf{g} = 0\bar{1}0$ , and this invisibility criterion is evidenced after the  
 7 profile analysis, as can be seen of Figure 6b.

8 These visibility/invisibility conditions were used to determine the nature of the different  
 9 defects. For a dislocation, knowing two conditions (two values of  $\mathbf{g}$ ) for which the dislocations  
 10 are invisible (i.e.,  $\mathbf{g}\cdot\mathbf{b}=\mathbf{0}$ ) enables determination of the Burgers vector. In this particular  
 11 example, the dislocations were classified depending on their visibility conditions.

12 In austenitic steel, the Burgers vector of the dislocations are expected to be of type  
 13  $\vec{\mathbf{b}} = \frac{1}{2} \langle 110 \rangle$ . Table 1 presents, for the different possible Burgers vector, the  
 14 visibility/invisibility conditions and the corresponding dislocations observed. Most observed  
 15 dislocations present a Burgers vector of type  $\vec{\mathbf{b}} = \frac{1}{2} [10\bar{1}]$ , as it was visible for the  $\mathbf{g} = 1\bar{1}\bar{1}$   
 16 and for the  $\mathbf{g} = \bar{1}\bar{1}1$  condition. This is in good agreement with the images presented on  
 17 Figure 2, for dislocations appearing as dots, *i.e.*, perpendicular to the surface. No dislocations  
 18 with Burgers vector of type  $\vec{\mathbf{b}} = \frac{1}{2} [110]$  or  $\vec{\mathbf{b}} = \frac{1}{2} [01\bar{1}]$  were found. One dislocation presents  
 19 visibility/invisibility conditions that corresponds to a Burgers vector of type  $\vec{\mathbf{b}} = \frac{1}{2} [1\bar{1}0]$  or of  
 20 type  $\vec{\mathbf{b}} = \frac{1}{2} [011]$ . Line dislocations, *i.e.* that are not perpendicular to the surface, were  
 21 detected to be visible only for the  $\mathbf{g} = 1\bar{1}\bar{1}$  condition, which do not corresponds to any  
 22 expected Burgers vector in the fcc system. In this case, the visibility of those dislocations in  
 23 the  $\mathbf{g} = 0\bar{1}0$  condition is questionable. Indeed, the line dislocations are visible on Figure 2c  
 24 but not on Figure 2d. This can be related to the crystallographic path followed during the  
 25 rotation series. The  $\mathbf{g} = 0\bar{1}0$  Kikuchi band is crossed relatively closed to the Zone Axis, and  
 26 some other higher indices planes might be crossed simultaneously that affects the contrast  
 27 between the defects and the matrix.

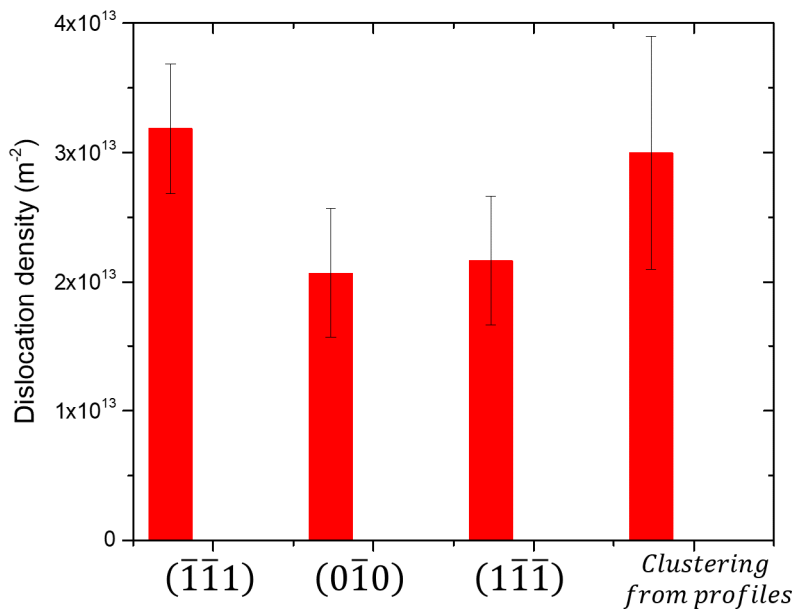
	$\frac{1}{2}[101]$	$\frac{1}{2}[10\bar{1}]$	$\frac{1}{2}[110]$ or $\frac{1}{2}[01\bar{1}]$	$\frac{1}{2}[1\bar{1}0]$ or $\frac{1}{2}[011]$	Other Burgers vector
$\mathbf{g} = \bar{1}\bar{1}1$	I	V	V	I	V
$\mathbf{g} = 0\bar{1}0$	I	I	V	V	I
$\mathbf{g} = 1\bar{1}\bar{1}$	I	V	I	V	I

Table 1 : Analysis of invisibility/visibility criteria.

1  
2

3 Once the defects were detected, the dislocation density was estimated using different  
4 methods. First, the density was estimated using a simple count method:  $\rho = N/S$ , where N is  
5 the number of defects and S is the analyzed area. Compared to the intercept method [17-18],  
6 or a 3D analysis using  $\rho = L/S.t$  (with  $t$  the analyzed thickness [11],  $L$  the total dislocation  
7 line and  $S$  the surface of the grain), this 2D counting method has the advantage not be  
8 affected by the uncertainty in the thickness of the analyzed volume nor by the appearing  
9 dislocation width. Moreover, the results are not dependent on the orientation of the  
10 dislocation relatively to the sample surface. The simple count method was performed  
11 manually on the images corresponding to the three diffracting conditions, whereas the  
12 number of detected dislocations from the clustering method were used to calculate the  
13 'clustering from profile' value. A dislocation density of  $(3.0 \pm 0.2) \times 10^{13} m^{-2}$  was estimated  
14 using the number of detected dislocations from the clustering method. It can be compared  
15 with the values obtained from a manual treatment on the images corresponding to the three  
16 diffracting conditions. For the  $\mathbf{g} = \bar{1}\bar{1}1$  condition, a rather similar value was measured  
17 directly on the image:  $(3.2 \pm 0.2) \times 10^{13} m^{-2}$ , while lower values were obtained for the  
18  $\mathbf{g} = 0\bar{1}0$  condition  $(2.0 \pm 20.2) \times 10^{13} m^{-2}$ , and for the  $\mathbf{g} = 1\bar{1}\bar{1}$  condition  $(2.2 \pm 0.5) \times$   
19  $10^{13} m^{-2}$  (see Figure 7). This is due to the fact that most dislocations are visible for the  $\mathbf{g} =$   
20  $\bar{1}\bar{1}1$  condition and are not visible for the two other conditions. Such a quite small dislocation  
21 density is in the range of what can be expected for this type of steel in its as-received  
22 condition [31].

1 In the present example, most of the dislocations were visible for the  $\mathbf{g} = \bar{1}\bar{1}1$  condition,  
 2 whereas some of them disappeared for the  $\mathbf{g} = 0\bar{1}0$  and  $\mathbf{g} = \bar{1}\bar{1}1$  conditions. This result  
 3 indicates that measurement of the dislocation density from a single image can lead to an  
 4 error up to a factor of 2, highlighting the key-importance to consider several two beam  
 5 conditions for a reliable dislocation density measurements, which is a tedious procedure  
 6 when performed on single images. Moreover, orientation of the specimen is required for  
 7 each two beam condition. When using a rotated image series, several two beam conditions  
 8 can be rapidly accessed at the same time. The direct analysis of the intensity profile rises  
 9 similar issues as manual image analysis for the evaluation of dislocation density. When  
 10 dislocation overlap, such as in the case of highly deformed samples ( $\rho > 10^{15}\text{m}^{-2}$ ), the  
 11 measurements will be biased. However, compared to a manual count, this method is fast and  
 12 efficient since no preliminar orientation of the sample is required.



13  
 14 *Figure 7: Comparison of dislocation density measurements from single image analysis and*  
 15 *clustering method analysis. A surface analysis using the simple count was performed and*  
 16 *compared with a 3D analysis.*

17  
 18 **Discussion**

19 The proposed methodology enables semi-automatic and quantitative dislocation  
 20 characterization, enabling efficient and fast dislocation density measurement, as the

1 measurements are simultaneously performed for several diffraction conditions. Moreover,  
2 the nature of the defects (Burgers vector, etc.) can be determined using a clustering  
3 procedure based on their visible/invisible criterion. The spatial resolution is that of the ECCI  
4 method, *i.e.*, 10–20 nm [11]. The classification of defects by type is beneficial for obtaining a  
5 better understanding of the mechanical properties of metallic materials as well as for the  
6 prediction of their mechanical behavior using constitutive laws.

7 This methodology was tested on a material presenting a small dislocation density ( $10^{13} \text{ m}^{-2}$ )  
8  $^2$ ), and therefore with grains that present almost no disorientation. Thus, the mean intensity  
9 profile of the matrix is the same whatever the position chosen in the grain. In the case of  
10 deformed samples, two problems might be encountered: the first one could be that the grain  
11 will present a disorientation leading to a variation in the mean intensity profile depending on  
12 the area selected. In that case, the data treatment should be adapted, and one solution could  
13 be to compare the intensity profile of a given pixel with the mean intensity profile around that  
14 given pixel. The second problem could be that, if the dislocation density is too high,  
15 dislocations will overlap on the images, making the measurement impossible, as for  
16 conventional ECCI image analysis. Therefore, this methodology is to date rather devoted to  
17 materials with dislocation density less than  $1-5 \times 10^{14} \text{ m}^{-2}$ .

18 Another limitation of the technique is that the crystallographic path cannot yet be  
19 selected. The methodology relies on a sufficient number of Kikuchi bands being crossed  
20 during the rotation. The crystallographic path depends mainly on two parameters: the  
21 accelerating voltage and tilt angle of the specimen. The accelerating voltage is selected to  
22 optimize the dislocation contrast; therefore, only the tilt of the sample can be adjusted. A  
23 statistical study is currently ongoing to determine the number of Kikuchi bands crossed for a  
24 random orientation. If, for a random orientation, the number of Kikuchi bands crossed is not  
25 sufficient, the development of a specific double tilt goniometer could allow more diffraction  
26 conditions to be accessed for the same grain.

27 Finally, the methodology is very dependent on the quality of the recorded image  
28 series. Important image drifts occurred during scanning at high magnification, which is  
29 required for dislocation imaging. Although some of the drift can be corrected by image  
30 analysis, more stable scanning systems are needed for further development of this method.

1 Distortion correction of the image using post-treatment methods could also limit the  
2 observed distortion.

3

#### 4 **Conclusion**

5 In this work, a new approach for measuring the dislocation density based on the CHORD  
6 method was presented. The methodology consists of acquiring a series of BSE images while  
7 rotating the sample under suitable conditions for dislocation imaging. After alignment of the  
8 entire image series, an intensity profile, which is a signature for the crystallographic  
9 orientation, is obtained for each pixel. For each two beam condition, if a defect is visible, the  
10 intensity of the pixels corresponding to this defect will differ from that of the matrix. Using a  
11 dedicated clustering algorithm, visibility and invisibility criteria are analyzed to determine the  
12 nature of each pixel (dislocation, noise or matrix). Subsequently, quantitative measurement of  
13 the dislocation density is possible, and the quantitative parameters of the defects (*e.g.*, the  
14 Burgers vector) can be deduced. This procedure can be applied regardless of the orientation  
15 of the considered grain. As the proposed method is fast and efficient, it enables the  
16 characterization of large representative areas through the realization of several series of  
17 rotation within the same sample.

18 In the future, this method could be applied for the simultaneous characterization of  
19 several grains of deformed alloys in a single step.

20

#### 21 **Data availability**

22 The raw/processed data will be made available on request.

23

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