# Influence of microscope settings on dislocation imaging in transmission forescattered electron imaging (t-FSEI)

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### Abstract

This work analyzes the influence of several microscope settings, namely, sample-forescattered electron detector (FSD) distance, and tilting conditions on the characteristics of the dislocation contrast imaged in transmission forescattered electron imaging (t-FSEI). The dislocation contrast behaviors of characteristic dislocation configurations of two Fe-based alloys, namely an α'martensitic (body-centered cubic, bcc) Fe-33Ni alloy (wt.%), and an austenitic (face-centered cubic, fcc) Fe-30Mn-6.5Al-0.3C alloy (wt.%) were investigated on thin foil samples by using different on-axis transmission Kikuchi diffraction (TKD) configurations, namely t-FSEI, brightfield (BF) t-FSEI and electron channeling contrast imaging (ECCI). The set-ups use transmission Kikuchi electron patterns to orient the crystal into controlled diffraction conditions. Imaging parameters such as dislocation contrast intensity and information depth are analyzed and compared to those obtained in the ECCI mode under the same microscope conditions. These effects are associated with the attenuation of Bragg scattering by high-angle scattering processes and the electron channeling mechanism, respectively. The experimental analysis sets the microscope settings for optimum dislocation imaging in t-FSEI.

**Keywords**: transmission forescattered electrons (t-FSE); Electron channeling contrast imaging; Dislocation imaging; scanning electron microscope (SEM); transmission Kikuchi diffraction (TKD)

#### 1-Introduction

The imaging of crystal defects in the scanning electron microscope (SEM) is commonly performed by the electron channeling contrast imaging (ECCI) technique [1-10]. ECC images of dislocations are formed by the acquisition of the modulated backscattered electron (BSE) signal associated with the localized lattice plane bending in the vicinity of a dislocation as the incident electron beam is scanned over the sample. Recently, a novel geometry for transmission Kikuchi diffraction (TKD) has been recently introduced (on-axis TKD) [11], where a phosphor screen oriented perpendicular to the incident beam is positioned below an electron transparent sample that is positioned at 0° tilt. The introduction of Si diodes attached to the camera head and placed below the thin foil allows the acquisition of the transmission forescattered electron (t-FSE) signal produced within the sample [12-15]. Similar to scanning transmission electron microscopy (STEM), t-FSE images with either strong crystallographic orientation contrast (bright-field forescattered electron ((BF) t-FSE) images) or atomic number contrast (dark-field forescattered electron ((DF) t-FSE) images) can be formed at small and large collection angles, respectively [12, 14, 16, 17]. Several studies have demonstrated the technical potential of these FSE imaging modes for microstructural characterization at the nano-scale [14, 16-18]. However, the characterization of crystal defects with the t-FSE signal in the SEM has been rather limited. So far, dislocation imaging with the t-FSE signal has been mainly performed by using a STEM detector [19-22]. These works have

demonstrated that dislocations and stacking faults can be characterized on thin foils in the SEM by using the t-FSE signal. In this work, we investigate the influence of several microscope settings, namely, sample-forescattered electron detector (FSD) distance, and tilting conditions on the dislocation contrast behavior imaged in transmission forescattered electron imaging (t-FSEI) by using an on-axis transmission Kikuchi diffraction (TKD) set-up. The dislocation contrast behavior of characteristic dislocation configurations of two Fe-based alloys, namely an α'-martensitic (body-centered cubic, bcc) Fe-33Ni alloy, and an austenitic (face-centered cubic, fcc) Fe-30Mn-6.5Al-0.3C alloy was investigated on thin foil-samples. Controlled diffraction conditions were calculated from the retrieved transmission Kikuchi electron patterns, as in [4, 7]. The influence of the microscope settings on the dislocation contrast was analyzed in terms of the scattering processes contributing to the formation of the t-FSE signal and the dislocation contrast. Imaging parameters such as the information depth and dislocation contrast intensity are analyzed and compared to those obtained in electron channeling contrast imaging (ECCI) under the same microscope conditions.

## 2-Materials and methods

#### 2.1-Material

Electron channeling contrast (ECC) and transmission forescattered electron (t-FSE) images of dislocations were acquired on thin foil samples of two Fe-alloys, namely, Fe-33Ni and Fe-30Mn-6.5Al-0.3C (wt.%). The crystal structures of the Fe-33Ni and Fe-30Mn-6.5Al-0.3C alloys are α'martensitic (body-centered cubic, bcc) and austenitic (face-centered cubic, fcc), respectively. Details of the processing of these alloys can be found in [23, 24]. The thin samples were prepared by two fabrication methods, namely, the twin-jet electrolytic polishing method by using a 10% perchloric acid at room temperature (Fe-33Ni alloy, wt.%) and the in-situ FIB lift-out method on a Scios 2 ThermoFisher Scientific dual-beam SEM system (Fe-30Mn-6.5Al-0.3C alloy, wt.%). Thinning of the FIB lamella was performed at an initial acceleration voltage of 30kV and a milling current of ~1000 pA on both sides of the foil followed by a final thinning procedure at 5 kV and 200 pA. The sample thickness at the region of interest of the Fe-33Ni alloy sample was determined using Electron Energy Loss Spectroscopy (EELS) in a JEOL JEM 2800 TEM operating at 200 kV with a convergence angle of 20 mrad and a collection angle of 42 mrad using the log-ratio technique [25]. The estimated sample thickness was  $220 \pm 10$  nm. The size of the lamella fabricated by FIB was about 20 μm x 20 μm x 100 nm. Before imaging, the specimen was plasma-cleaned using an XEI Evactron 25 (XEI Scientific, Inc., Redwood City, USA) de-contaminator. ECCI and t-FSEI were performed on a field emission gun scanning electron microscope (FEG-SEM) Zeiss Sigma (Zeiss, Oberkochen, Germany) equipped with an on-axis transmission Kikuchi diffraction (TKD) system that consists of a Bruker e-FlashHD EBSD (Bruker, Billerica, MA, USA) camera mounted on a Bruker OPTIMUS<sup>TM</sup> detector head. The detector head consists of a phosphor screen and three forescattered electron detectors (FSD) with a size of  $5.2 \times 4.4 \text{ mm}^2$  for collecting the transmission forescattered electron (t-FSE) signal produced within the sample. TKD measurements and ECC/t-FSE images were collected at 30 kV acceleration voltage with an aperture of  $60 \mu m$ . The microscope was run in the "high current" mode with a probe current of  $\sim 8.5 \text{ nA}$ . Tilting experiments were performed on a 5-axis motorized eucentric stage (x, y, z, tilt, rotation) with an x/y/z step resolution of 1  $\mu m$  and tilt/rotation step resolution of  $0.1^{\circ}$ . SEM images were taken at  $1024 \times 1024$  pixels resolution with reduced digital noise (5 minutes per scan).

The SEM set-ups used in this study, namely transmission forescattered electron imaging (t-FSEI) (a), bright-field (BF) t-FSEI (b), and electron channeling contrast imaging (ECCI) (c), are shown in the sketch of Fig. 1. t-FSE images were acquired by a forescattered electron detector (FSD) located in the middle of the detector head and placed beneath the thin sample. The (BF) t-FSEI mode corresponds to the acquisition of t-FSE images by placing the FSD normal to the optic axis [14, 17, 26]. In the current SEM configuration, the minimum collection angle  $\alpha_l$  and the maximum collection angle  $\alpha_2$  as a function of the optical axis-FSD distance ( $DD_2$ ) are defined as:

$$\alpha_1 = \tan^{-1} \left( \frac{DD_2}{DD_1} \right) \tag{1}$$

$$\alpha_2 = \tan^{-1}\left(\frac{DD_2 + a}{DD_1}\right) \tag{2}$$

where  $DD_I$  is the sample-FSD distance along the optical axis ( $DD_I = 18$  mm) and a is the FSD width ( $\alpha = 5.2$  mm). The effective collection angle,  $\Delta \alpha$ , is defined as  $\Delta \alpha = \alpha_2 - \alpha_1$ . In the ECCI mode (Fig. 1c), ECC images of dislocations were acquired by a solid-state four-quadrant backscattered electron detector (BSED) that collects the BSE signal emitted from the upper surface of the thin foil. The ECCI set-up is characterized by a low-tilt configuration at a 7 mm working distance. The sample-BSED configuration is the same as that used in the conventional EBSDbased ECCI set-up [27, 28]. In the present set-up, the phosphor screen is set underneath the beam to directly acquire transmission Kikuchi electron patterns (TKEPs) of the imaged area. Following the approach described in [4, 27], the Euler angles determined from the collected TKEPs are used calculate the corresponding electron channeling patterns (ECPs), and hence, the channeling/diffraction conditions used to form the ECC/t-FSE images at a given tilting condition. It is relevant to indicate that the region of the thin foil where TKEPs are formed may be different from that of the formation of the BSE/t-FSE signals. TKEPs are formed in a region of about 10 – 100 nm from the bottom surface of a thin foil [17, 29, 30] whereas BSE/t-FSE signals are typically generated within the whole thickness of a thin foil [31-35]. Accordingly, the on-axis transmission Kikuchi diffraction (TKD) set-ups shown in Fig. 1 are limited to the imaging of dislocations on

thin foils containing a constant crystallographic orientation along the sample thickness. For this, the structural size of the analyzed region along the sample thickness must be considerably larger than the thin foil thickness.

## 3-Results and discussion

# 3.1-Effect of detector distance on the dislocation contrast

This section presents the analysis of the effect of the optical axis-FSD distance, DD<sub>2</sub>, on the intensity of the dislocation contrast imaged by the transmission forescattered electron signal (t-FSE). Fig. 2 shows a series of t-FSE images of dislocations taken at different values of DD<sub>2</sub> corresponding to a range of maximum collection angle,  $\alpha_2$ , between 140 mrad and 815 mrad. Ranges of maximum and minimum collection angles,  $\alpha_1$  and  $\alpha_2$ , calculated with equations (1) and (2) for different values of DD<sub>2</sub> are shown in Table 1. The t-FSE images were acquired on a thinfoil sample of a Fe-33Ni alloy (thickness of 220 ± 10 nm) and taken under two-beam diffraction conditions using the diffraction vector (2 1 1) with w > 0 (w: deviation parameter). As a comparison, the (BF) t-FSE image taken with  $\alpha_2 = 145$  mrad is shown in Fig. 2(a). The series of t-FSE images reveal two main characteristics, namely, the visibility of the dislocation contrast within a large range of  $\alpha_2$  ( $\sim$  785 mrad), and the attenuation of the intensity of the dislocation contrast with the

detector distance, and hence  $\alpha_2$ . Specifically, when  $\alpha_2 < 500$  mrad, dislocations exhibit a strong contrast characterized by a dark sharp contrast over a bright background similar to that imaged in (BF)-TEM. When  $\alpha_2 > 500$  mrad, the dislocation contrast becomes weaker and vanishes at  $\alpha_2 \sim$ 815 mrad. This effect is analyzed in Fig. 3. This figure plots the variation of the transmitted electron yield, TEY, (ratio of the number of collected transmitted electrons by the total number of electrons), dislocation contrast intensity,  $I_{dc}$ , and intensity of the t-FSE signal of the matrix,  $I_{matrix}$ , as a function of  $\alpha_2$ . TEY was calculated from the Monte Carlo simulations of electron trajectories produced by scattering elastic scattering and inelastic events approximated by a mean energy loss model between two elastic scattering events by the software Casino version 3.3 [36-38]. The simulations were obtained for an accelerating voltage of 30 kV and a beam diameter of 3 nm, which is approximately equal to that used experimentally. One million electron trajectories were simulated to decrease the statistical variation inherent in the Monte Carlo method.  $I_{dc}$  was measured from the intensity profile measured across the dislocation line indicated by an arrow in Fig. 2.  $I_{dc}$ is defined as  $I_{dc} = (I_{max} - I_{min})/(I_{max} + I_{min})$ , where  $I_{max}$  and  $I_{min}$  correspond to the highest and lowest values of the intensity profile of the t-FSE signal across a dislocation.  $I_{matrix}$  is estimated as the average value of the pixel intensity of a matrix region free of dislocations.

Fig. 3 shows that the experimentally determined attenuation of  $I_{matrix}$  with  $\alpha_2$  follows a similar trend to the variation of TEY with  $\alpha_2$  calculated by Monte Carlo simulations. This means that the total number of collected forescattered electrons decreases with the optical axis-FSD distance  $(DD_2)$  and hence, the intensity of the t-FSE signal is attenuated. Interestingly, the variation of  $I_{dc}$ with  $\alpha_2$  reveals several features of the influence of the detector distance on the dislocation contrast. The present results indicate that in the current configuration ( $DD_1 = 18 \text{ mm}$ ), a strong dislocation contrast ( $I_{dc} \sim 0.10 - 0.15$ ) is imaged on t-FSE images by placing the FSD at a small optical axis-FSD distance, DD<sub>2</sub>, between 0 mm (BF-mode) and 4 mm. It corresponds to a maximum collection angle range ( $\alpha_2$ ) of 525 mrad. With increasing DD<sub>2</sub>, the dislocation contrast becomes weaker ( $I_{dc}$ < 0.10 for  $525 < \alpha_2 < 785$  mrad) and it vanishes at  $DD_2 = 14$  mm ( $\alpha_2 > 815$  mrad). The observed dependence of the dislocation contrast on the detector distance can be qualitatively explained as follows. The collected t-FSE signal consists of small-angle scattered electrons (Bragg scattering) and high-angle scattered electrons (Rutherford scattering and thermal diffuse scattering) [39]. At small values of  $\alpha_2$ , the diffracted signal strongly contributes to the collected t-FSE signal resulting in a strong dislocation contrast. At increased values of  $\alpha_2$ , the t-FSE signal is gradually formed by high-angle scattered electrons due to the attenuation of Bragg scattering by thermal vibrations. At large values of  $\alpha_2$ , the t-FSE signal is dominated by thermal diffuse scattered electrons and exhibits a Z<sup>2</sup>-dependence (Z-contrast) [40-42]. Several works have reported Z-contrast on t-FSE images

acquired at large collection angles [12, 26]. The angular dependences of the scattering processes contributing to the t-FSE signal results in the attenuation of  $I_{dc}$  with  $\alpha_2$  resulting in a gradual smearing out of the dislocation contrast. This effect has been reported on a thin foil sample of a Zr alloy containing Nb particles using a similar on-axis transmission Kikuchi diffraction (TKD) configuration than that shown in Fig. 1(a) [26]. This study shows that under similar microscope conditions to those used in the present study, diffraction contrast vanishes at large maximum collection angles ( $\alpha_2 > 760$  mrad). According to Jesson and Pennycook [43, 44], the minimum collection angle for Z-contrast scales with  $\lambda/\Delta R$ , where  $\lambda$  is the electron wavelength and  $\Delta R$  is the distance between neighboring atomic columns. The smaller  $\alpha_2$  reported in the Zr sample where diffraction contrast vanishes (Zr:  $\alpha_2 > 760$  mrad; Fe33-Ni:  $\alpha_2 > 815$  mrad) can be associated with the larger atomic plane spacing of the Zr sample used to form the t-FSE images.

## 3.2-Influence of channeling conditions on the dislocation contrast

The influence of channeling conditions on the intensity of the dislocation contrast was evaluated on (BF) t-FSE images of the deformation band (DB) structure of a Fe-30Mn-6.5Al-0.3C alloy (wt.%) deformed at -196 °C. The deformation band structure is formed by an inhomogeneous deformation process, as described in [23]. Fig. 4 shows two examples showing the effect of the

deviation parameter, w, on the dislocation contrast of the dislocation configuration of a deformation band displayed on ECC images (a, c) and the corresponding (BF) t-FSE images (b, d) taken under two-beam conditions using the diffraction vector (2 4 0). ECC/(BF) t-FSE images (a, b) and (c, d) were taken under w < 0 and w > 0 channeling/diffraction conditions, respectively. The diffraction conditions were set by tracking the modulation of the BSE signal with w, as in [4]. Due to the low intensity of the BSE signal produced by the thin sample (BSE yield is strongly dependent on the sample thickness), dislocation contrast is only visible on the (BF) t-FSE images. In a thicker thin-foil (thickness of ~ 220 nm), dislocation contrast is visible on a ECC image (Fig. 6(b)). The deformation band (DB) is characterized by a localized dislocation configuration of ~1.5 μm wide that is formed by closely spaced dislocation configurations lying on non-coplanar {111} slip systems. The dislocation configuration is visible as dark compact layers over a bright background. The deformation band is delimited by a boundary region of about 0.2 - 0.4 µm wide containing a high localized plastic deformation.

The ECC/(BF) t-FSE images reveal a strong channeling effect on the intensity of the BSE/t-FSE signal. As expected, the ECC images exhibit a strong dependence of the BSE signal on the deviation parameter, w, which is associated with the channeling mechanism [4, 45]. Interestingly, the (BF) t-FSE images also reveal a strong channeling effect on the intensity of the t-FSE signal. Fig. 5 plots the intensity profiles of the t-FSE signal measured across the same dislocation

configuration (indicated by an arrow in Fig. 4) imaged under w > 0 and w < 0 conditions, respectively. The width of the dislocation configuration (measured from the width of the profile at half peak height) estimated from both profiles is similar, i.e. about 65 nm. However, under w > 0conditions, the t-FSE signal associated with the crystal matrix,  $I_{t-FSE}^{matrix}$ , exhibits a stronger intensity  $(I_{t-FSE}^{matrix} = 175 \pm 25 \text{ (gray value)})$  than that under w < 0 conditions  $(I_{t-FSE}^{matrix} = 75 \pm 10 \text{ (gray value)})$ , Figs. 4(b, d). As the intensity of the t-FSE signal associated with the dislocation configuration is roughly constant under the current channeling conditions, the channeling effect results in an enhanced dislocation contrast. Defining the dislocation contrast intensity,  $I_{dc}$ , as  $I_{dc} = (I_{t-FSE}^{matrix})$  $I_{t-FSE}$  //  $I_{t-FSE}^{dis}$  +  $I_{t-FSE}^{dis}$ , where  $I_{t-FSE}^{matrix}$  and  $I_{t-FSE}^{dis}$  correspond to the average intensities of the t-FSE signal of the crystal matrix and that of the matrix region containing a dislocation, respectively, it yields  $I_{dc} = 0.45$  (w > 0) and  $I_{dc} = 0.3$  (w < 0). The present findings indicate that channeling effects play a significant effect in the intensity of the dislocation contrast in (BF)-tFSEI. The observed channeling effect is similar to that occurring on the forward-scattered beam in TEM [46-48]. The effect is associated with the inelastic and incoherent scattering of the transmitted FSE signal as the primary beam travels along the lattice crystal (channeling mechanism), resulting in the modulation of the t-FSE signal with the deviation parameter, w. The effect of w on the t-FSE signal is reverted to that on the BSE signal due to the characteristic modulation of the BSE/t-FSE signals with w. The channeling effect is expected to be visible in thick samples (thickness > 100nm) and contributes to the intensity of the dislocation contrast displayed on (BF) t-FSE images.

# 3.3-Information depth

As ECCI is a relevant SEM imaging mode of dislocations, we have comparatively analyzed the information depths in (BF) t-FSEI and ECCI on a thin sample of Fe-33Ni alloy (wt.%) (sample thickness of  $220 \pm 10$  nm). The information depths were analyzed through the comparison of the dislocation contrast imaged by (BF) t-FSEI and ECCI under the same channeling conditions. (BF) t-FSE and ECC images were acquired by the set-ups shown in Fig. 1(b) and Fig. 1(c), respectively. Fig. 6 shows an example of a (BF) t-FSE image (a) and an ECC image (b) of the dislocation configuration in the interior of a α' martensite plate taken under two-beam diffraction conditions using the diffraction vector (-2 1 1), with w > 0. The images display a projection of two sets of dislocation lines. Most of the dislocation lines are straight and a few curved dislocation lines are visible, which indicates the presence of a local stress field. Considering the bcc-structure of  $\alpha'$ martensite, the observed dislocations correspond to <111>-type screw dislocations [24]. Accordingly, the Burgers vector is parallel to the dislocation line direction. As expected, the dislocation contrast displayed on the (BF) t-FSE image is inverted to that shown on the ECC image, as shown in the intensity profiles of Figs. 6(c), 6(d). These plots were acquired across the same dislocation line (indicated by an arrow in Figs. 6(a), 6(b)). Although the absolute value of the dislocation contrast intensity displayed on these images can not be compared due to the different detector settings that influence the absolute intensity value [31], the reversion of the contrast is measurable. The plots also show that the dislocation feature width, y, imaged on the (BF) t-FSE/ECC images is similar ( $y \sim 31$  nm measured on the (BF) t-FSE image;  $y \sim 33$  nm measured on the ECC image).

Fig. 7 shows the analysis of the visible dislocation lines imaged on the (BF) t-FSE image and ECC image. Blue lines correspond to dislocation lines that are imaged in the same configuration in both imaging modes. Red lines correspond to dislocation lines that are imaged in a different dislocation configuration. The analysis reveals that the dislocation configuration imaged in the two imaging modes is nearly identical. Only a few dislocation lines were imaged in a slightly different configuration. The analysis indicates that in the analyzed thin foil, the dislocation contrast displayed in both imaging modes, i.e. (BF) t-FSEI and ECCI, originates within the same sample thickness. As the t-FSE signal is generated within the whole sample thickness, it suggests that under the current microscope conditions, the ECCI information depth,  $d_{ECCI}$  is at least the thickness of the analyzed area, i.e.  $d_{ECCI} > 220 \pm 10$  nm.  $d_{ECCI}$  of a given material can be estimated by the estimation of the attenuation of the intensity of the primary electron Bloch wave when traveling through a crystal [49]. It yields  $d_{ECCI} \sim 5 \xi_g$ , where  $\xi_g$  is the extinction distance for a given diffraction vector and electron beam energy. This approach has been used to estimate  $d_{ECCI}$ at acceleration voltages of 20 - 30 kV in polycrystalline materials containing crystal defects [2, 5,

22, 50, 51]. Under the current (-2 1 1) channeling conditions,  $\xi_{211} \sim 50$  nm for bcc-Fe at 100 KeV [47, 48]). Assuming an energy correction where the ratio of the relativistic factors is considered [47], we obtain  $\xi_{211} \sim 45$  nm, and hence,  $d_{ECCI} \sim 225$  nm. This value agrees with the range of  $d_{ECCI}$ experimentally determined from the observations of the dislocation configurations. Accordingly, our analysis indicates that in the present thin-foil sample and under the current microscope conditions, the ECCI information depth,  $d_{ECCI}$  is the same as the t-FSEI information depth,  $d_{t-FSEI}$ , resulting in an identical imaged dislocation configuration (same number of visible dislocation lines and similar dislocation feature width) that is displayed with a comparatively reverted contrast. However, if the ECC images are formed using a diffraction vector with small  $\xi_g$  (for instance,  $\xi_{110}$ ~ 25 nm for bcc-Fe at 100 KeV),  $d_{ECCI} < d_{t-FSEI}$  and hence, the dislocation configuration displayed on ECC images will be different to that displayed on the corresponding (BF) t-FSE images taken under the same diffraction conditions. This effect may be useful for understanding 3D effects on the dislocation configuration imaged in ECCI and (BF) t-FSEI.

## 4-Conclusions

This work analyzes the influence of several microscope settings, namely, sample-forescattered electron detector (FSD) distance, and tilting conditions on the dislocation contrast behavior in transmission forescattered electron imaging (t-FSEI). These effects were investigated on the

dislocation configuration of thin-foil samples of two Fe-based alloys, namely an  $\alpha$ '-martensitic (body-centered cubic, bcc) Fe-33Ni alloy (wt.%), and an austenitic (face-centered cubic, fcc) Fe-30Mn-6.5Al-0.3C alloy (wt.%) on several on-axis transmission Kikuchi diffraction (TKD) setups. The following conclusions can be drawn:

-The optical axis-FSD distance,  $DD_2$ , has a strong influence on the intensity of the dislocation contrast imaged in t-FSEI. In the current microscope set-up, a strong dislocation contrast ( $I_{dc} \sim 0.10 - 0.15$ ) is imaged on t-FSE images by placing the FSD at a small optical axis-FSD distance,  $DD_2$ , between 0 mm (BF-mode) and 4 mm. With increasing  $DD_2$ , the dislocation contrast becomes weaker ( $I_{dc} < 0.10$  for  $525 < \alpha_2 < 785$  mrad) and it vanishes at  $DD_2 = 14$  mm ( $\alpha_2 > 815$  mrad). This effect is attributed to the attenuation of Bragg scattering by high-angle scattering processes resulting in the attenuation of the dislocation contrast with  $\alpha_2$ .

-Channeling effects play a significant role in the dislocation contrast intensity in (BF) t-FSEI. We find that the dislocation contrast intensity enhances under w > 0 conditions. This effect is associated with the channeling effect generated as the primary beam travels along the lattice crystal resulting in a modulated t-FSE signal. Comparatively, the influence of the channeling effect on the t-FSE signal is reverted to that on the BSE signal due to the characteristic modulation of the BSE and t-FSE signals with the deviation parameter, w.

-Experimental determinations of the ECCI information depth,  $d_{ECCL}$  and t-FSEI information depth,  $d_{t-FSEI}$ , through the analysis of the imaged dislocation configuration show that under specific microscope conditions ((2 1 1) diffraction conditions and 30 kV),  $d_{ECCL} = d_{t-FSEI}$ . This results in an identical imaged dislocation configuration (same number of visible dislocation lines and similar dislocation feature width) that is displayed with a comparatively reverted contrast. We expect that under diffraction conditions where the extinction distance is small (for instance,  $\xi_{II0} \sim 25$  nm for bcc-Fe at 100 KeV), the dislocation configuration imaged in ECCI will be different from that displayed in the corresponding (BF) t-FSE image. This effect may be useful for understanding 3D effects on the dislocation configuration imaged in ECCI and (BF) t-FSEI.

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**Table 1.** Values of  $\alpha_1$  (minimum collection angle),  $\alpha_2$  (maximum collection angle), and  $\Delta\alpha$  (effective collection angle) as a function of DD<sub>2</sub> (detector distance).

DD <sub>2</sub> (mm)	0	4	6	8	10	12	14
$\alpha_1$ (mrad)	-140	220	320	420	510	615	660
α <sub>2</sub> (mrad)	140	475	555	625	695	785	815
Δα (mrad)	280	250	235	205	185	170	155

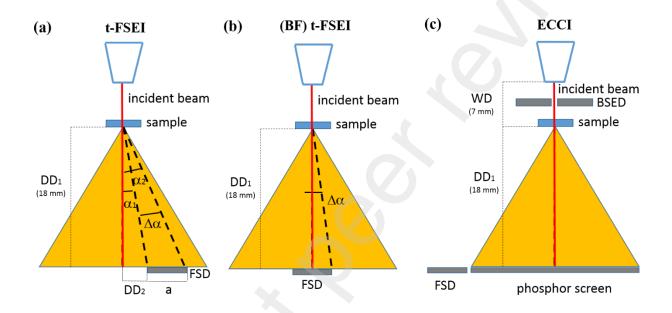
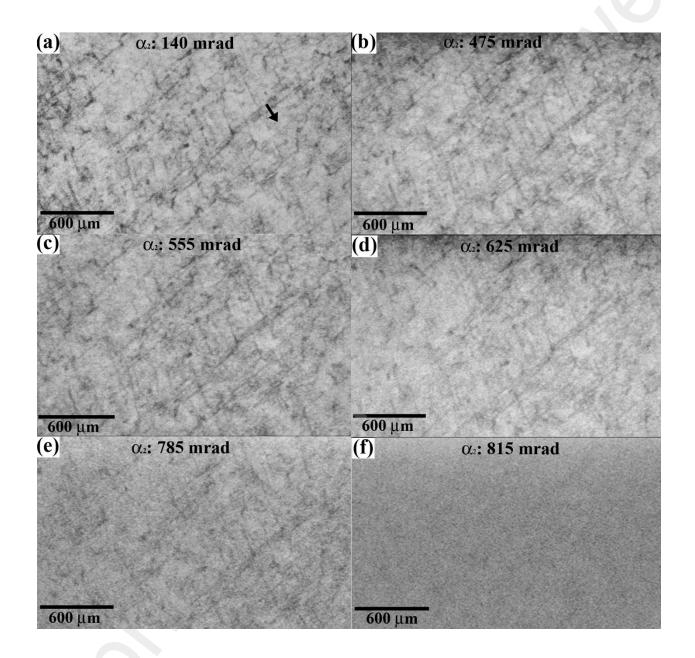


Figure 1. Schematic of the SEM set-ups. (a): Transmission forescattered electron imaging (t-FSEI); (b): Bright-field transmitted forescattered electron imaging ((BF) t-FSEI); (c): Electron channeling contrast imaging (ECCI).  $DD_I$ : sample-FSD distance along the optical axis.  $DD_2$ : detector distance. a: FSD width.  $\alpha_I$ : minimum collection angle.  $\alpha_2$ : maximum collection angle.  $\Delta \alpha$ : effective collection angle. BSED: backscattered electron detector. FSED: forescattered electron detector.



**Figure 2.** Influence of the maximum collection angle ( $\alpha_2$ ) on the dislocation contrast imaged in t-FSEI using the set-up shown in Fig. 1(a). The t-FSE images were collected on a thin foil of a Fe-33Ni alloy (thickness of  $220 \pm 10$  nm) and taken under two-beam diffraction conditions using g (2 1 1) with w > 0 (w: deviation parameter).

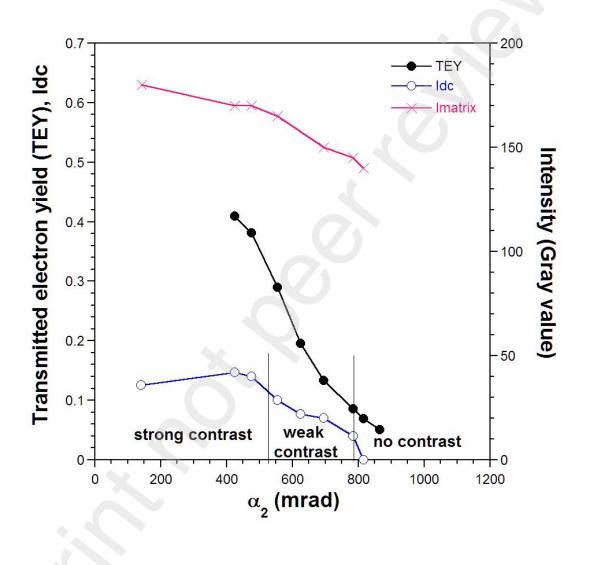
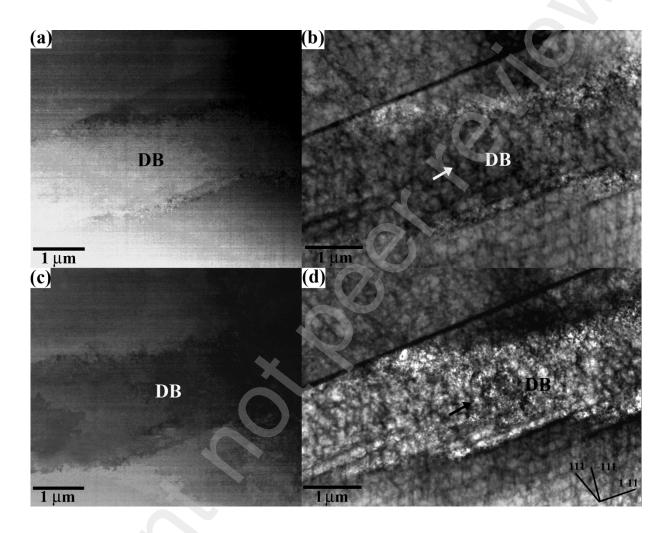
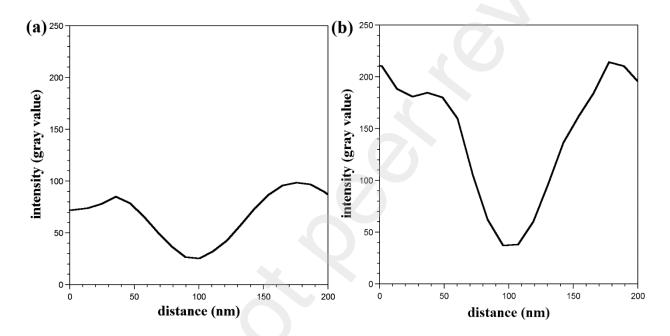


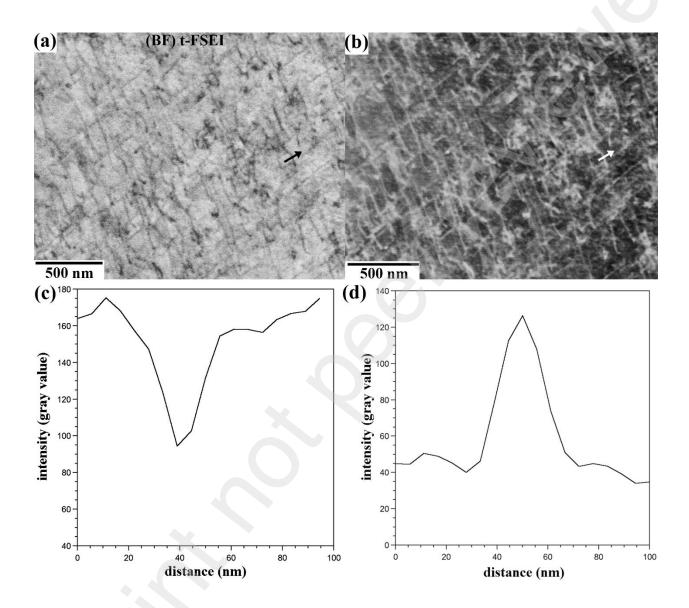
Figure 3. Variation of transmitted electron yield (TEY), dislocation contrast intensity,  $I_{dc}$ , and intensity of the t-FSE signal of the matrix,  $I_{matrix}$ , as a function of maximum collection angle,  $\alpha_2$ .



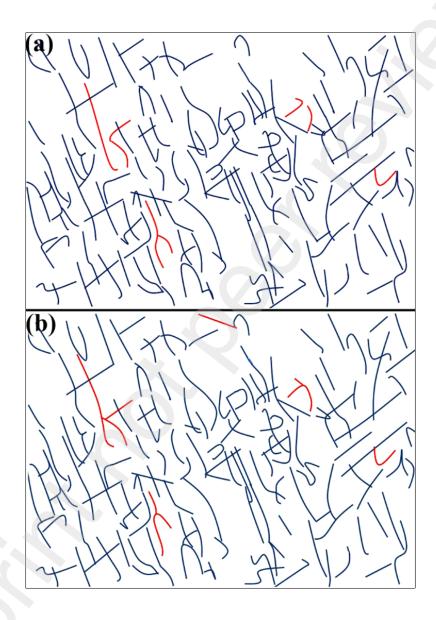
**Figure 4**. Influence of the channeling conditions on the ECC images (a, c) and (BF) t-FSE images of the dislocation configuration of a deformation band structure of a Fe-30Mn-6.5Al-0.3C alloy deformed at -196 °C. The ECC/(BF) t-FSE images were taken under two-beam conditions using g (2 4 0). (a, b): w < 0 conditions. (c, d): w > 0 conditions. DB: Deformation band.



**Figure 5.** Intensity profiles of the t-FSE signal measured across the dislocation configuration indicated in Fig. 4. (a): w < 0 conditions. (b): w > 0 conditions.



**Figure 6**. (BF) t-FSE image (a) and ECC image (b) of the dislocation configuration in the interior of a  $\alpha'$  martensite plate taken under the same two-beam diffraction conditions using the diffraction vector (-2 1 1) with w > 0. (c, d): Intensity profiles across the dislocation line indicated by an arrow in (a) and (b).



**Figure 7**. Analysis of the visible dislocation lines imaged in Fig. 4. (a): (BF) t-FSEI; (b): ECCI. Blue lines correspond to dislocation lines showing the same configuration imaged in both imaging modes. Red lines correspond to dislocation lines with a different imaged dislocation configuration.

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