Article

Modeling dislocation contrasts obtained by accurate-Electron Channeling Contrast Imaging for characterizing deformation mechanisms in bulk materials

Hana KRIAA [1], Antoine GUITTON [1,2] and Nabila MALOUFI [1,2,*]

[1] Université de Lorraine - CNRS - Arts et Métiers ParisTech – LEM3, 7 rue Félix Savart, 57070 Metz, France; hana.kriaa@univ-lorraine.fr (H.K.); antoine.guitton@univ-lorraine.fr (A.G.); nabila.maloufi@univ-lorraine.fr (N.M.)

[2] Laboratory of Excellence on Design of Alloy Metals for low-mAss Structures (DAMAS) – Université de Lorraine, 57073 Metz, France.

* Correspondence: nabila.maloufi@univ-lorraine.fr; Tel.: +33 372 747 865

Received: date; Accepted: date; Published: date

Abstract: Electron Channeling Contrast Imaging (ECCI) is becoming a powerful tool in Materials Science such as for characterizing deformation defects. Dislocations observed by ECCI in Scanning Electron Microscope, exhibit several features depending on the crystal orientation relative to the incident beam (white/black line on a dark/bright background). In order to bring new insights concerning these contrasts, we report an original theoretical approach based on the dynamical diffraction theory. Our calculations led, for the first time, to an explicit formulation of the backscattered intensity as function of various physical and practical parameters governing the experiment. Intensity profiles are modeled for dislocations parallel to the sample surface for different channeling conditions. All theoretical predictions are consistent with experimental results.

Keywords: ECCI, dislocation contrast, modeled intensity profiles, invisibility criteria, dynamical theory of electron diffraction.

1. Introduction

The Electron Channeling Contrast Imaging (ECCI) takes advantage from the strong dependency of the BackScattered Electrons (BSE) signal on the orientation of the incident beam with the lattice planes due to the electron channeling mechanism [1]. Therefore, any slight local distortion of the crystal lattice, produced for instance by a dislocation leads to a BSE intensity (I_{BSE}) modulation, thus generating several contrasts such as bright line on a dark background [2] or black line on bright background [3].

For understanding the origin of the dislocation contrasts obtained by ECCI, the two-beam dynamical diffraction theory was adapted from the Transmission Electron Microscopy (TEM) [4] [5]. Briefly, the electron beams are described, inside the crystal, by a superposition of Bloch waves. The different inelastic scattering events are divided into two categories: those scattered through angles less than 90° (forming the forward scattering wave) and those scattered through angles greater than 90° (forming the backscattered wave) [6]. In the multiple scattering model electrons can be removed from the forward scattering wave to the backscattered one and vice-versa. In order to simulate the I_{BSE} profiles for both perfect and imperfect crystal, Spencer *et al.* [7] and Wilkinson *et al.* [6, 8, 9] used this Bloch wave-based model. They showed that for the perfect crystal, the simulated profiles exhibit

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the main experimental features of the channeling pattern: bright band and dark edges. The same approach was also used by Reimer [10, 11] for a perfect crystal where the multiple scatterings are treated as incoherent. These different approaches, were extended to the case of an imperfect crystal containing a dislocation [6-9] or a stacking fault [12]. Despite their contribution to the theory of the defects electron channeling contrasts [7-12], detailed calculations leading to an analytical expression of BSE signal as a function of experimental parameters, is still missing. Furthermore, in most cases [7,8] theoretical results were not confronted with the experiments. This can be illustrated from the dislocation profiles calculated for Bragg condition, which exhibit an extra-pic of I_{BSE} not observed experimentally [7, 8].

To go deeper in the understanding of the observed channeling contrast of dislocations, we propose an easier way for modeling the I_{BSE} as a function of physical parameters either relative to the material or governing the ECCI experiment. Our theoretical results show a good agreement with the experiments for several diffraction conditions.

In a crystal, the electronic wave function is solution of the time independent Schrödinger's equation and is given by [11]:

$$\Psi(\mathbf{r}) = \sum_j \varepsilon^{(j)} \sum_g C_g^{(j)} e^{[2\pi i \left(\mathbf{k_0}^{(j)}+\mathbf{g}\right)\cdot\mathbf{r}]} e^{[-2\pi q^{(j)} z]} \quad (1)$$

The index j refers to the jth wave, $\varepsilon^{(j)}$ are the excitation amplitudes of the Bloch wave $\psi^{(j)}$, $C_g^{(j)}$ are the amplitudes of the diffracted waves with a wave vector $\mathbf{k_g^{(j)}} = \mathbf{k_0^{(j)}} + \mathbf{g}$, where $\mathbf{k_0^{(j)}}$ is the wave vector of the jth primary wave and $\mathbf{g}$ is the diffraction vector. $\mathbf{r}$ is the spatial position vector at which the electron intensity is evaluated. The second factor of equation (1) contains the absorption parameter $q^{(j)}$ expressing the exponential attenuation of the wave amplitude with increasing depth z.

In order to determine the different coefficient of the Bloch wave function, presented in equation (1), Reimer used the two-beam condition *i.e.* only one set of lattice planes are in channeling condition. Hence, the total BSE signal of a slice of a thickness dz, located at a depth z is given by [11]:

$$\frac{d\eta}{dz} = N\sigma_B \{\psi\psi^* + (1 - \sum_j \left|C_0^{(j)}\right|^2 e^{[-4\pi q^{(j)} z]})\} \quad (2)$$

N is the atom number per unit of volume, σ_B is the backscattering cross-section through angles larger than 90° and $\psi\psi^*$ is the probability for the Bloch wave to be backscattered at a depth z. The last terms (in parentheses) in equation (2) describes the electrons that are removed from the Bloch wave field by scattering before reaching the slice dz.

The BSE coefficient $\eta_{O.C.}$ is, then, obtained from the integration of equation (2) in the total interaction depth from z=0 to $z \to \infty$ (labelled $\Delta\eta$ in Reimer's model). O.C. indicates that only the total BSE intensity due to orientation contrast is calculated, while the contributions due to atomic number and to the surface inclination are not considered [11]:

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$$\eta_{O.C.} = \frac{N\sigma_B}{4\pi}\,\xi_0'\left(-\frac{s\,\xi_g+\frac{\xi_0'}{\xi_g'}}{1+(s\,\xi_g)^2-(\frac{\xi_0'}{\xi_g'})^2}+\frac{\omega}{1+(s\,\xi_g)^2+[(1+(s\,\xi_g)^2)(\frac{\xi_0'}{\xi_g'})^2]}\right) \quad (3)1$$

Equation (3) corresponds to the variation of the BSE intensity for a perfect crystal i.e. the intensity profile of an isolated pseudo-Kikuchi band [7,11,13]. Some of the parameters of equation (3) will be defined later in the text.

2. Our theoretical approach for BSE intensity calculation for an imperfect crystal

If we consider a column located at a position x away from a dislocation, at a position x=0 and depth $z=z_D$ (where z_D is the mean depth of the dislocation), the distortion of the lattice planes nearby the dislocation does not depend on z but only on x and it is given by $\left(\frac{\partial \mathbf{R}}{\partial z}\right)_{z=z_D}$ (**R** is the displacement field of the crystalline planes) [14].

Therefore, for calculating the I_{BSE} in the case of an imperfect crystal containing a dislocation parallel to the sample surface, independently of the depth z, we take into account a new deviation parameter s' written by:

$$s' = s + s_D \quad \text{where} \quad s_D = \mathbf{g}\cdot\left(\frac{\partial \mathbf{R}}{\partial z}\right)_{z=z_D} \quad (4)$$

s is the deviation from the exact Bragg position in the perfect crystal, which can be experimentally measured [3]. The scalar product $\mathbf{g}\cdot\left(\frac{\partial \mathbf{R}}{\partial z}\right)_{z=z_D}$ represents the supplementary deviation, s_D due to the variation of the incidence angle between the primary beam and the distorted crystalline planes near the dislocation core. Far from the dislocation, the crystal is considered as perfect. The planes are not distorted and the deviation s_D becomes zero. Consequently, to take into account the presence of the defect, we substitute s by s' in the expression of $\eta_{O.C.}$ for a perfect crystal (in equation 3, which does not depend on z). We obtain:

$$\eta_{O.C.} = \frac{N\sigma_B}{4\pi}\,\xi_0'\left(-\frac{(s+s_D(x)_{z=z_D})\xi_g+\frac{\xi_0'}{\xi_g'}}{1+((s+s_D(x)_{z=z_D})\xi_g)^2-(\frac{\xi_0'}{\xi_g'})^2}+\frac{(s+s_D(x)_{z=z_D})\xi_g}{1+((s+s_D(x)_{z=z_D})\xi_g)^2+[(1+((s+s_D(x)_{z=z_D})\xi_g)^2(\frac{\xi_0'}{\xi_g'})^2]}\right) \quad (5)$$

This allow to study the variation of the I_{BSE} as a function of x (distance x away from the dislocation core). Where, the contrast associated to a dislocation is described by s_D (containing all the effect of **R**).

2.1. Screw dislocation

Figure 1 shows a screw dislocation parallel to the surface of a bulk sample and located at a depth z_D. This defect is characterized by a Burgers vector **b** and a line direction **u**. At a distance x away from the dislocation core (in x=0), the crystal plane is deformed. The displacement field $\mathbf{R_{screw}}$ is then defined in polar coordinate (ß) as follows [15]:

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1 Note that in the book of Reimer [11], equation (3) contains an error: it is written 2π to the denominator instead of 4π.

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$$\mathbf{R}_{\mathbf{screw}} = \frac{\mathbf{b}\,ß}{2\pi} = \frac{\mathbf{b}}{2\pi}\,\tan^{-1}\left(\frac{z-z_D}{x}\right) \quad (6)$$

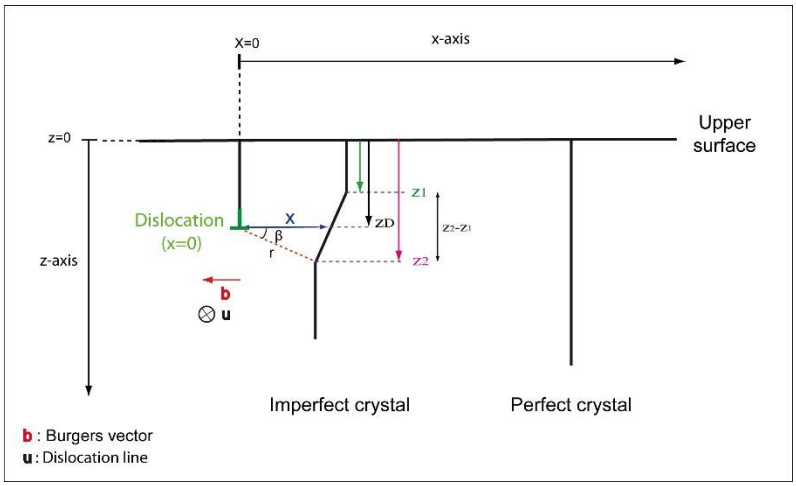

Figure 1. Schematic of an edge dislocation parallel to the surface and located at a depth z_D. Deformed planes, perpendicular to the surface, are at a distance x away from the dislocation core.

The derivative of $\mathbf{R}_{\mathbf{screw}}$ with respect to the depth z, at a turning point ($z = z_D$), is given by:

$$\left(\frac{d\mathbf{R}_{\mathbf{screw}}}{dz}\right)_{z=z_D} = \left(\frac{\mathbf{b}}{2\pi x\left(1+\left(\frac{z-z_D}{x}\right)^2\right)}\right)_{z=z_D} = \frac{\mathbf{b}}{2\pi x} \quad (7)$$

Based on this reasoning, the substitution of equation (7) in equation (5) allows us to obtain the following expression of $\eta_{0.C.}$:

$$\eta_{0.C.} = \frac{N\sigma_B}{4\pi}\,\xi_0'\left(-\frac{(s+\frac{\mathbf{g}\cdot\mathbf{b}}{2\pi x})\xi_g+\frac{\xi_0'}{\xi_g'}}{1+((s+\frac{\mathbf{g}\cdot\mathbf{b}}{2\pi x})\xi_g)^2-(\frac{\xi_0'}{\xi_g'})^2}+\frac{(s+\frac{\mathbf{g}\cdot\mathbf{b}}{2\pi x})\xi_g}{1+((s+\frac{\mathbf{g}\cdot\mathbf{b}}{2\pi x})\xi_g)^2+\{[1+((s+\frac{\mathbf{g}\cdot\mathbf{b}}{2\pi x})\xi_g)^2](\frac{\xi_0'}{\xi_g'})^2\}}\right) \quad (8)$$

Equation (8) gives the variation of the BSE signal as a function of the distance x and the experimental parameters such as the deviation s and the diffraction vector **g**.

It should be noted that in this paper, we show profiles modeled in the case of aluminum, where the used parameters are: acceleration voltage E=20 kV, **g**= (220), the extinction distance ξ_g=50 nm, absorption lengths ξ_0'=140 nm and ξ_g'=600 nm [11]. It should also be mentioned that in all modeled profiles the background level will be taken as reference (at the zero of the ordinate axis). All negative values then correspond to lower BSE intensities than the background level.

2.1.1. Deviation parameter s=0

The theoretical intensity profiles calculated from equation (8), in the case of a screw dislocation, for the diffraction conditions s=0 and ±**g** are represented in Figure 2. Their corresponding experimental ECC micrographs are also showed (Figures 2a' and b'). The **g** and **s** vectors are, respectively, determined experimentally through the pseudo-band indexation of the HR-SACP (High Resolution Selected Area Channeling Pattern) assisted by EBSD experiment [2, 3].

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For both +**g** and –**g** diffractions, the dislocation profiles (Figures 2.a and b) are anti-symmetric: a hollow and a peak corresponding to the black and white sides of the dislocation respectively (Figures 2a' and b'). Moreover, in the case of –**g**, the extrema are inverted compared to those observed for +**g**: the peak becomes hollow and vice versa.

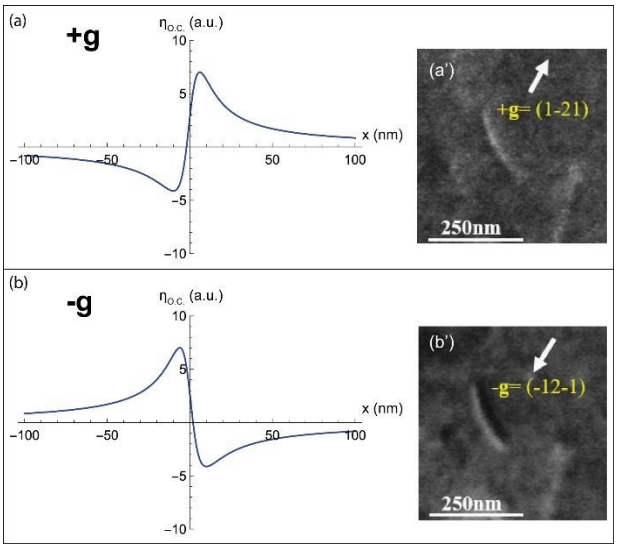

Figure 2. I_{BSE} profiles modeled, for a screw dislocation parallel to the surface, with a deviation parameter s=0 for the diffractions (a) +**g** and (b) –**g** with their corresponding experimental ECC micrographs (a') and (b').

Such theoretical results reveals that at Bragg position, a screw dislocation generates a BSE image with black/white sides, which reverse with the inversion of the sign of **g**. Therefore, equation (8) is in good agreement with the experimental observations already reported in literature [3,7].

2.1.2. Deviation parameter s>0

The I_{BSE} profiles calculated by equation (8) with a deviation parameter slightly positive (s=0.01 nm^{-1}) are represented in Figures 3a and b for the +**g** and –**g** diffractions respectively. In this condition (s>0), both ±**g** dislocation profiles present one intensity peak only. This is in agreement with the experimental ECC micrographs shown in Figures 3a' and b': bright line on dark background. Note also that the maximum intensity does not coincide with the exact position of the dislocation core (x=0 nm) but it is at x≈±4 nm: it is displaced from one side of the dislocation position to the other side when changing from +**g** to –**g**. Such result is analogous to that obtained in TEM and can be used to characterize a dislocation configuration consisting of two parallel dislocations such as dipole [3, 16].

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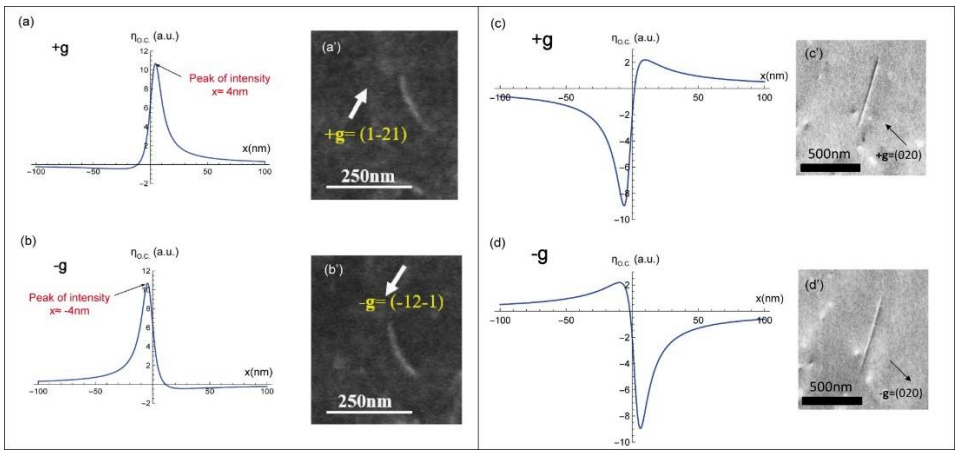

Figure 3. I_{BSE} profiles modeled, for a screw dislocation parallel to the surface, with s>0 and s<0 for (a) (c) **+g** and (b) (d) **–g** with their corresponding experimental ECC micrographs (a'), (b'), (c') and (d').

2.1.3. Deviation parameter s<0

The I_{BSE} profiles calculated from our theoretical model for a slightly negative deviation parameters (s=-0.01 nm^{-1}) and **±g** diffraction conditions are represented in Figure . For the diffraction **+g**, the curve contains a deep hollow and a peak corresponding to the black and white dislocations sides respectively (Figure 3c'). This contrast is inverted with the inversion of the sign of **g** (Figures 3d and d'). For s<0, the BSE signal emitted from the zone of interest is high: bright background.

2.2. Edge dislocation

Similar to screw dislocation, an edge dislocation parallel to the surface and located at a depth z_D produces a local deformation of the crystalline planes nearby its core. Such distortion is described by its displacement field, written in polar coordinate, as follows [15]:

$$\mathbf{R}_{edge}=\frac{\mathbf{b}}{2\pi}\left[ß+\frac{\sin 2ß}{2(1-\nu)}\right]+\frac{\mathbf{b}\times\mathbf{u}}{2\pi}\left[\frac{1-2\nu}{2(1-\nu)}\ln|r|+\frac{\cos 2ß}{4(1-\nu)}\right] \quad (9)$$

ν is the Poisson's ratio, **u** is the dislocation line direction and r is the polar coordinate. Where ß and r are given by:

$$ß=\tan^{-1}\left(\frac{z-z_D}{x}\right) \text{ and } r=\frac{x}{\cos ß} \quad (10)$$

From equations (9) and (10), $\mathbf{R}_{edge}$ can be expressed as a function of the distance x away from the dislocation. The new deviation parameter s' is then:

$$s' = s + \mathbf{g}\cdot\frac{d\mathbf{R}_{edge}}{dz})_{z=z_D} \quad (11)$$

The presence of an edge dislocation in the crystal can also be highlighted, analytically, by the insertion of equation (11) in equation (5). Hence the calculated theoretical profiles are similar to that obtained for a screw dislocation. For the diffraction **+g**, at Bragg condition (s=0), the modeled curves are characterized by a maximum and a minimum of I_{BSE}. The edge dislocation generates, then, a white/black contrast. However, for s>0, profile presents only a single peak consistent with experimental observations. This maximum of intensity is situated at a position x≈-6 nm away from the dislocation core. Concerning the case of s<0, the I_{BSE} profile show a hollow with a slight peak. All

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profiles are also reversed, following the inversion of the **g** sign regardless of the deviation parameter s.

2.3. Extinction criteria

Furthermore, for both screw and edge dislocations, considering the extinction criteria $\mathbf{g}\cdot\mathbf{b}=0$ and $\mathbf{g}\cdot\mathbf{b}\times\mathbf{u}=0$ in our equation leads to a null BSE yield ($\eta_{O.C.}=0$ a.u in Figure 4a). Regarding the edge dislocation, the $\mathbf{b}\times\mathbf{u}$ term in equation (9) becomes null when $z= z_D$. Nevertheless, the position of the dislocation is located in the $[z_1, z_2]$ range (see Figure 1), therefore the $\mathbf{b}\times\mathbf{u}$ term is not null. For $\mathbf{g}\cdot\mathbf{b}=0$ and $\mathbf{g}\cdot\mathbf{b}\times\mathbf{u}\neq0$, in the $[z_1, z_2]$ range except z_D, the calculated profile for an edge dislocation displays a low intensity peak $\eta_{O.C.}\approx2,7$ (a.u) with respect to the background level) surrounded by two hollows. Such residual contrast (Figure 4b) is characteristic of an edge dislocation observed by TEM under these diffraction conditions [17].

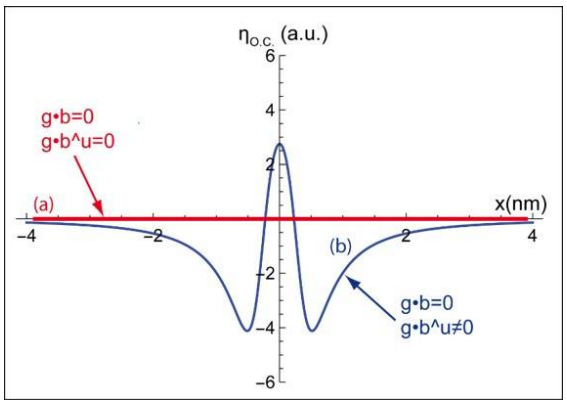

Figure 4. IBSE profiles modeled for the extinction conditions: (a) $\mathbf{g}\cdot\mathbf{b}=0$, $\mathbf{g}\cdot\mathbf{b}\times\mathbf{u}=0$ and (b) $\mathbf{g}\cdot\mathbf{b}=0$, $\mathbf{g}\cdot\mathbf{b}\times\mathbf{u}\neq0$.

2.4. Quantitative comparisons with experimental profiles

In this part, for each deviation parameter: s>0, s<0 and s=0, an average profile is calculated from 50 experimental dislocation profiles and fitted by equation (5). The results are illustrated by Figures 5a, b and c respectively.

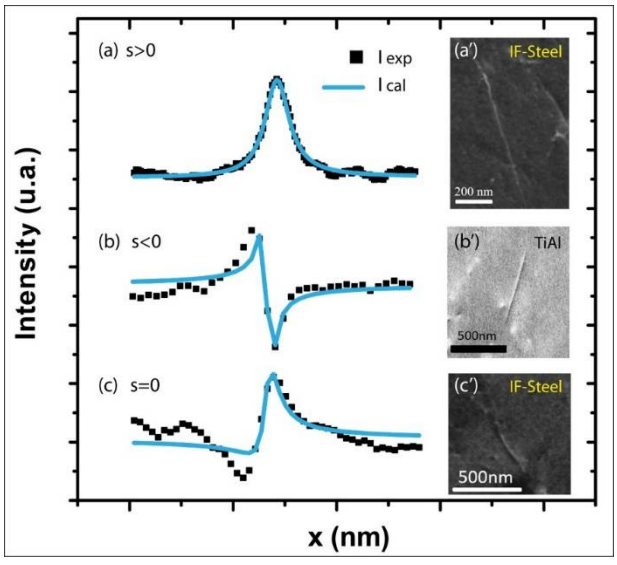

Figure 5. Fitted (blue line) and experimental (black squares) I_{BSE} profiles and their corresponding ECC micrograph obtained for (a, a') s>0, (b, b') s<0 and (c, c') s=0 respectively.

As can be seen, the best fits are obtained for s>0 (the correlation coefficient χ^2=2) and s<0 (χ^2=8.7). While for s=0, the general features of the curve are well modeled but the correlation coefficient is higher: χ^2=16.4. At Bragg condition because of the strong interaction between the electron beam and the crystal atoms [18], dynamical effects are magnified and the diffracted intensity is higher enough to excite neighboring reflections. Then the successively and coherently produced beams interfere with each other. The "n" beam approach must thus be considered to better report the experimental results. Besides, in our calculations multiple scattering was treated incoherently.

Nevertheless, the fitted profiles provide, among other parameters, reasonable orders of magnitude of the physical parameters ξ_g, $\xi_0^{'}$ and $\xi_g^{'}$ for different deviation parameters and materials (IF-steel: Figure 5a, a', c and c' TiAl: Figures 5b and b'). Furthermore, the obtained parameters are in good agreement with the values reported in the literature [11]. Such as the case of IF-steel: ξ_g=9,4±1,3 nm ; $\xi_0^{'}$=170,4±36,7 nm ; $\xi_g^{'}$=177,7±38,3 nm.

3. Conclusions

In this paper an original theoretical model based on the Bloch wave approach of the dynamical diffraction theory was developed for modeling I_{BSE} around dislocations without resorting to numerical methods. An analytical formula of the BSE signal as a function of the different physical parameters governing the ECCI experiment is proposed for the first time to our knowledge. The BSE contrast profiles, produced by screw and edge dislocations parallel to the sample surface, display the same appearance for the diffraction conditions. For a deviation parameter s=0 (Bragg condition) and s<0, the theoretical BSE curves show hollow and peak of intensity corresponding to the black and white dislocation sides repectively. The inversion of the **g** leads to the profile inversion (hollow becomes peak and vice versa). For s>0, the bright dislocation contrast is envisaged in the modeled profile by the intensity peak. This latter (dislocation image) do not coincide with the exact dislocation position (x=0) and it is displaced to the opposite side when the **g** is reversed. Moreover, our theoretical model confirms the use of the invisibility criteria in ECCI.

The good agreement between the theoretical and experimental results was also confirmed by adjusting the BSE intensity profiles. Hence, deduced values for the physical parameters ξ_g, the extinction distance and $\xi_0^{'}$ and $\xi_g^{'}$, the absorption lengths are consistent with the literature.

Because the use of ECCI is becoming widespread for the defects characterization in bulk material in SEM, we provide a usable formula of the BSE intensity produced by dislocations. Furthermore, our approach can be extended to other defects. Now ECCI is mature for exploring new horizons in Materials Science.

Author Contributions: All ECCI observations were performed by H.K. H.K., A.G. and N.M. performed the theoretical interpretations. H.K. wrote the main manuscript text. All the authors participate in the discussion.

Conflicts of Interest: The authors declare no conflict of interest.

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