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From Electron Tomography of Dislocations to Field Dislocation Mechanics: Application to Olivine

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3 **From Electron Tomography of Dislocations to Field Dislocation Mechanics:**
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5 **Application to Olivine**
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1 **Abstract**

2 We propose a new procedure to extract information from electron tomography and use them as
3 an input in a field dislocation mechanics. Dislocation electron tomography is an experimental
4 technique that provides three-dimensional information on dislocation lines and Burgers vectors
5 within a thin foil. The characterized 3D dislocation lines are used to construct the spatial
6 distribution of the equivalent Nye dislocation density tensor. The model dislocation lattice
7 incompatibility equation and stress balance equation are solved with a spectral code based on
8 fast Fourier transform algorithms. As an output of the model, one obtains the three-dimensional
9 distribution of mechanical fields, such as strains, rotations, stresses, resolved shear stresses and
10 energy, inside the material. To assess the potential of the method, we consider two regions from
11 a previously compressed olivine sample. Our results reveal significant local variations in local
12 stress fields and resolved shear stresses in various slip systems, which can impact the strong
13 plastic anisotropy of olivine and the activation of different dislocation slip systems. It also
14 evidences the built-up of kinematic hardening down to the nanometre scale.

15
16 **Keywords:** Dislocations; Electron Tomography; Mechanics; Plasticity; Olivine

17 18 **1 Introduction**

19 Making the link between plastic flow and the dynamics of crystal defects (dislocations) that
20 causes it is a formidable challenge that requires the description of their collective behaviour at
21 the mesoscopic scale [1]. To this end, mesoscopic simulations such as discrete dislocation
22 dynamics (DDD) [2, 3] represent a powerful analysis tool that should be complementary to
23 experimental observations of dislocations. Complementary experimental/modelling analysis of
24 dislocation microstructures can bring valuable insights about elementary deformation
25 mechanisms. Transmission electron microscopy is usually the experimental tool of choice for

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3 26 this kind of investigation since it allows a fine characterization of dislocations and their
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5 27 interactions. However, the understanding of the 3D microstructures is difficult since we only
6
7 28 have access to 2D projections and the tilt capabilities are physically limited in microscopes.
8
9 29 Dislocation Electron Tomography (DET) has been developed to overcome these limitations.
10
11 30 The basic principle as originally proposed by Barnard *et al.* (2006) [4] is to perfectly orient the
12
13 31 diffraction vector used to image the dislocations parallel to the tilt axis, maintaining at best as
14
15 32 possible the Bragg deviation parameter. This technique has been applied in numerous studies
16
17 33 as analyses of dislocation microstructures near cracks [5, 6], characterization of helical
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19 34 configurations of dislocations [7-9], analyses of specific interactions of dislocations [10],
20
21 35 studies of interactions of dislocations with grain and sub-grain boundaries [11-14], analyses of
22
23 36 dislocation microstructures in a carbide [15] and minerals [16-19]. The DET has been the
24
25 37 subject of several recent reviews [20-23]. Recent developments include reconstructions from
26
27 38 few projected images using black & white contrasts of dislocations [9, 19] or “stereo-pair”
28
29 39 method [24-27], machine-learning reconstruction [28] and 4D analysis [9]. In the present
30
31 40 contribution, we propose to use electron tomography to get access to the line directions of the
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33 41 dislocation segments. The association of these line directions with their corresponding Burgers
34
35 42 vectors allows the construction of the Nye dislocation density tensor at any point in the
36
37 43 reconstructed space. Subsequently, the Nye dislocation density tensor leads up to the prediction
38
39 44 of the internal mechanical fields (strain, stress, energy, etc.) through the use of a Field
40
41 45 Dislocation Mechanics (FDM) model that allows to build a bridge between real dislocation
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43 46 microstructures and continuum mechanics. To illustrate the potential of this DET/FDM analysis
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45 47 method, we consider here as a benchmark the characterization of dislocation networks in an
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47 48 experimentally deformed olivine sample. We will consider in this work two different areas from
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49 49 a deformed single crystal. We reveal for this complex material (limited number of dislocation
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51 50 slip systems, strong plastic anisotropy) a three-dimensional stress state characterized by
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3 51 significant spatial variations and long-range elastic interactions between dislocations. The
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5 52 resulting distribution of dislocation driving stresses allows suggesting possible active slip
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7 53 systems and the strong heterogeneity of distributions shows that the history of dislocation
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9 54 dynamics and the associated build-up of kinematic hardening can be captured with the proposed
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11 55 method.
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18 57 **2 Materials and deformation experiments**

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20 58 This study was carried out on an olivine sample (PoEM9) experimentally deformed by
21
22 59 Demouchy *et al.* (2013) [29] which had already been the focus of TEM microstructural
23
24 60 investigations [10, 17]. These axial compression experiments were carried out on a cylinder
25
26 61 (length 6.32 mm; diameter 4.19 mm) specimen extracted from a large, gem-quality, single
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28 62 crystal of San Carlos olivine ($(\text{Mg}_{0.91}\text{Fe}_{0.09}\text{Ni}_{0.003})_2\text{SiO}_4$). The sample PoEM9 [29], was
29
30 63 deformed along [502] in a high-resolution gas-medium high-pressure apparatus [30] at 806 °C,
31
32 64 with a constant strain rate of $5.1 \times 10^{-5} \text{ s}^{-1}$, under a gaseous (Ar) confining pressure of 300 MPa.
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34 65 The finite strain reached was $\epsilon_{\text{Total}} = 10.1 \%$ (obtained post-mortem) and the maximum
35
36 66 differential stress was 754 MPa.
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44 68 **3 Methods**

45 69 ***3.1 Transmission electron microscopy***

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48 70 A complete characterization of the dislocations must include not only the Burgers vectors, but
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50 71 also the geometry of the lines and their habit planes.

51 72 ***3.1.a Indexation of Burgers vectors***

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54 73 In the earlies 1980s, Ishida *et al.* [31] have shown that the product of the Burgers vectors \mathbf{b} of
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56 74 a dislocation and of the diffraction vector \mathbf{g} used to perform the weak-beam dark-field (WBDF)
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58 75 image is equal to the number of thickness fringes which ends at the extremity of this considered

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3 76 dislocation (the direction of the ending thickness fringes is linked to the sign of the Burgers
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5 77 vector [32]).

6
7
8 78 Figure 1 gives an example of a typical WBDF micrograph of PoEM9 where the diffraction
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10 79 vector \mathbf{g} : 004 was used to image dislocations (this reflection was chosen as it has the highest
11
12 80 structure factor in olivine). The $\mathbf{g} \cdot \mathbf{b}$ product is equal to ± 4 with a $\pm[001]$ Burgers vector. This
13
14 81 number clearly corresponds to the number of terminating thickness fringes seen on figure 1b
15
16 82 and 1c.

17
18
19 83 Figure 1

20
21 84 Asymmetry of the contrast intensity of dislocation extremities is linked to the sign of the
22
23 85 Burgers vectors. This information can be compared to the one obtained from the directions of
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25 86 terminating thickness fringes (Figure 2). Taking all these indices into account, we were able to
26
27 87 determine most of the Burgers vector signs.

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29
30 88 Figure 2

31 32 33 89 **3.1.b Dislocation geometry characterization**

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35 90 The electron tomography technique entails acquiring a tilt-series and then utilizing a
36
37 91 reconstruction algorithm to obtain a corresponding three-dimensional (3D) model of the
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39 92 microstructure. As outlined in prior studies [8, 16], the principal impediment to performing
40
41 93 electron tomography of dislocations is to maintain a diffraction contrast (which is highly
42
43 94 sensitive to the thin foil orientation) constant across the tilt-series. In order to address this issue,
44
45 95 the diffraction vector, used to image dislocations, must be precisely aligned with the principal
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47 96 axis of the sample-holder. Furthermore, a slight precession of the electron beam is applied to
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49 97 homogenize the background contrasts (including the presence of thickness fringes) and the
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51 98 dislocation contrasts (oscillating contrasts of dislocations which cross the thin foil thickness)
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53 99 [16, 33]. To enhance the dislocation contrasts, a numerical filter has been employed in ImageJ
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58 100 to adjust the contrast. Reconstructions are then conducted with the weighted back-projection
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3 101 algorithm [34], while dislocations are directly redrawn into the reconstructed volume using
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5 102 markers, facilitated by the Chimera-UCSF software [8, 9, 15, 18-20, 35], to attain precisely the
6
7 103 3D coordinates of the dislocation core images.

104 *3.2 Nye tensor and elastic field distribution in an anisotropic elastic material*

105 The Field Dislocation Mechanics (FDM) model employed here [36] introduces discrete
106 dislocation lines, as characterized by tomography, in the form of equivalent continuous
107 dislocation density spatial distributions on a regular three-dimensional grid made of voxels. As
108 an output, the model provides the equilibrated stress field in the voxelized thin foil volume, as
109 well as the associated elastic displacements, strains, and rotations. The characteristics of
110 dislocations, namely their line directions and their Burgers vectors, are introduced through the
111 Nye dislocation tensor in the model [37]. The Nye tensor α is a second-order tensor with
112 components $\alpha_{ij} = B_i t_j$ in the cartesian coordinate system ($\mathbf{e}_1, \mathbf{e}_2, \mathbf{e}_3$) aligned with the thin foil
113 frame. The quantity B_i is a length of Burgers vector per unit surface and can be written $B_i =$
114 $nb/\Delta_S \mathbf{e}_i$, where n is the number (not necessarily an integer) of dislocations of Burgers vector
115 magnitude b along the direction \mathbf{e}_i , and Δ_S is the spatial resolution surface. The vector \mathbf{t} is the
116 local dislocation line unit vector. For instance, an edge (respectively screw) dislocation with
117 line along the \mathbf{e}_3 direction and with Burgers vector along the \mathbf{e}_1 (respectively \mathbf{e}_3) direction
118 corresponds to an edge (respectively screw) dislocation density α_{13} (respectively α_{33}). A mixed
119 dislocation would be made of both densities. In practice, any three-dimensional dislocation line
120 or segment can be transferred into an equivalent Nye dislocation density distribution in a
121 volume. In the case of a regular grid made of voxels to be used later for fast Fourier transform
122 (FFT) based numerical calculation of elastic fields, we use the recent method proposed by
123 Bertin [38] to transform discrete dislocation segments into equivalent dislocation densities
124 properly assigned on voxels. Note that a similar approach was also proposed very recently [39].

1
2
3 125 The method used is briefly presented below, the reader is referred to reference [38] for more
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5 126 details.

7
8 127 Figure 3

9
10 128 Figure 3 illustrates a part of a discrete dislocation line composed of three straight segments
11
12 129 shown in red in the figure. The figure shows a two-dimensional setup for clarity, but the
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14 130 calculation is performed in three dimensions. The second (middle) dislocation segment is
15
16 131 considered here and shown as a bold red line. It is defined by its starting point, \mathbf{x}_a , and its ending
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18 132 point, \mathbf{x}_b , following the direction of the segment line vector \mathbf{t} . The thin dashed black lines
19
20 133 delimit the grid voxels used in the numerical algorithm used to solve FDM equations. The voxel
21
22 134 size is H_1 , H_2 , and H_3 in the \mathbf{x}_1 , \mathbf{x}_2 , and \mathbf{x}_3 directions. The position of the centre of voxel d
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24 135 considered in the figure is given by its position vector \mathbf{x}_d . Its volume is denoted $\Omega_d =$
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26 136 $H_1 \times H_2 \times H_3 = H^3$ where we chose that $H_1 = H_2 = H_3 = H$. For the voxel d , a surrounding
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28 137 box of dimension $2H_1 \times 2H_2 \times 2H_3$ is centered at point \mathbf{x}_d and is shown with purple solid lines
29
30 138 in the figure. If a part of a given dislocation segment falls within this box, then a Nye dislocation
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32 139 density associated with this segment will be attributed to this voxel, as follows. If start and/or
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34 140 end points $\mathbf{x}_a/\mathbf{x}_b$ are outside the box, the dislocation segment is cropped to new start and/or end
35
36 141 points $\mathbf{x}_a'/\mathbf{x}_b'$. If they are inside, they remain as they are. Now consider a point \mathbf{x} along the
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38 142 dislocation segment. We define the vector $\mathbf{R}_d = \mathbf{x}_d - \mathbf{x}$, which brings points \mathbf{x} on the segment to
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40 143 the centre of voxel \mathbf{x}_d . The unit vector $\mathbf{t} = (\mathbf{x}_b - \mathbf{x}_a) / \|\mathbf{x}_b - \mathbf{x}_a\|$ is the segment tangent vector. The
41
42 144 distance from the segment supporting line to point \mathbf{x}_d is denoted d . It is the norm of the vector
43
44 145 $\mathbf{d} = \mathbf{R}_d - (\mathbf{R}_d \cdot \mathbf{t})\mathbf{t} = \mathbf{x}_d - \mathbf{x}_0$. The vector \mathbf{d} starts at point \mathbf{x}_0 on the segment supporting line and ends at
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46 146 point \mathbf{x}_d . With this, one can parametrize the position \mathbf{x} along the dislocation segment as $\mathbf{x} = \mathbf{x}_0 + s\mathbf{t}$,
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48 147 where the scalar value $s \in [s_a, s_b]$. The two bounds s_a and s_b shown in the figure (s_a' and/or s_b' if
49
50 148 the segment is cropped) are $s_a = (\mathbf{x}_a - \mathbf{x}_0) \cdot \mathbf{t}$ and $s_b = (\mathbf{x}_b - \mathbf{x}_0) \cdot \mathbf{t}$. From these definitions, one can finally

149 parametrize the position vector $\mathbf{R}_d = \mathbf{x}_d - \mathbf{x}$ as $\mathbf{R}_d = \mathbf{d} \cdot \mathbf{st}$. The dislocation density at the voxel d due
 150 to a dislocation segment is given by the following line integral

$$151 \quad \alpha_{ij}(\mathbf{x}_d) = \frac{b_i t_j}{\Omega_d} \int_{x_a}^{x_b} S(\mathbf{x}_d - \mathbf{x}) dL(\mathbf{x}) = \frac{b_i t_j}{\Omega_d} I(\mathbf{x}_d), \quad (1)$$

152 where the weight function S is taken to be a Cloud-In-Cell function defined by

$$153 \quad S(\mathbf{x}_d - \mathbf{x}) = \begin{cases} \prod_{i=1}^3 \left(1 - \frac{|\mathbf{x}_i^d - \mathbf{x}_i|}{H_i}\right) & \text{if } |\mathbf{x}_i^d - \mathbf{x}_i| < H_i, \\ 0 & \text{otherwise.} \end{cases} \quad (2)$$

154 The integral $I(\mathbf{x}_d)$ can be numerically evaluated as

$$155 \quad I(\mathbf{x}_d) = (s^b - s^a) - A_1 - A_2 - A_3 + B_{12} + B_{13} + B_{23} - C_{123}, \quad (3)$$

156 with the terms

$$157 \quad \begin{cases} A_i = \frac{1}{H_i} \left| \left[d_i s - \frac{1}{2} t_i s^2 \right]_a^b \right|, \\ B_{ij} = \frac{1}{H_i H_j} \left| \left[d_i d_j s - \frac{1}{2} (d_i t_j + t_i d_j) s^2 + \frac{1}{3} t_i t_j s^3 \right]_a^b \right|, \\ C_{ijk} = \frac{1}{H_i H_j H_k} \left| \left[d_i d_j d_k s - \frac{1}{2} (t_i d_j d_k + d_i t_j d_k + d_i d_j t_k) s^2 \right. \right. \\ \left. \left. + \frac{1}{3} (d_i t_j t_k + t_i d_j t_k + t_i t_j d_k) s^3 - \frac{1}{4} t_i t_j t_k s^4 \right]_a^b \right|. \end{cases} \quad (4)$$

158 The domain $[s_a, s_b]$ is divided into portions $[a, b]$ on which the sign of $\prod_{i=1}^n (d_i - st_i)$ does not
 159 change. For each voxel d , the dislocation density is the sum of all densities due to all dislocation
 160 segments that cross the surrounding box shown in figure 3. The process is repeated for all voxels
 161 forming the simulation volume. Given an initial Nye dislocation density in a volume, we are
 162 now interested in numerically estimating the associated internal stress field within a small strain
 163 anisotropic elasticity mechanical framework. We rely on the FDM model [36]. We denote by
 164 \mathbf{U}_e the elastic distortion of a three-dimensional body containing dislocations. In the absence of
 165 dislocations, the distortion would be the gradient of the elastic displacement vector \mathbf{u}_e , that is
 166 $\mathbf{U}_e = \mathbf{grad}(\mathbf{u}_e)$. However, it is not true anymore in the presence of dislocations due to
 167 discontinuities in the elastic displacement field, i.e., the Burgers vectors of dislocations. As

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3 168 such, the elastic distortion must contain an incompatible, non-gradient, curl part ($\mathbf{curl}(\boldsymbol{\chi})$),
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5 169 which is related to the Nye tensor. The elastic distortion is thus written as

$$170 \quad \mathbf{U}_e = \mathbf{U}_e^{\parallel} + \mathbf{U}_e^{\perp} = \mathbf{grad}(\mathbf{u}_e) + \mathbf{curl}(\boldsymbol{\chi}). \quad (5)$$

171 The elastic distortion resumes to the compatible, gradient part, in the absence of dislocations,
172 while the incompatible, curl part, is related to the Nye tensor through the incompatibility
173 equation

$$174 \quad \mathbf{curl}(\mathbf{U}_e^{\perp}) = \boldsymbol{\alpha} = \mathbf{curl}(\mathbf{curl}(\boldsymbol{\chi})), \quad (6)$$

175 Where the curl operator removes the compatible part of the elastic distortion. For a given
176 distribution of Nye dislocation density in a volume, the elastic incompatible distortion is
177 obtained from the solution of equation (6). Then, the compatible elastic distortion is generally
178 nonzero and ensures the balance of stresses inside the volume. That writes

$$179 \quad \mathbf{div}(\boldsymbol{\sigma}) = \mathbf{div}(\mathbf{C} : \mathbf{U}_e) = \mathbf{div}(\mathbf{C} : (\mathbf{U}_e^{\parallel} + \mathbf{U}_e^{\perp})) = \mathbf{0}, \quad (7)$$

180 where $\boldsymbol{\sigma}$ is the Cauchy stress tensor and \mathbf{C} is the fourth-order elastic moduli tensor. The above
181 equation can be rearranged as

$$182 \quad \mathbf{div}(\mathbf{C} : \mathbf{grad}(\mathbf{u}_e)) = -\mathbf{div}(\mathbf{C} : (\mathbf{U}_e^{\perp})), \quad (8)$$

183 and solved for the elastic displacement field \mathbf{u}_e . Once the elastic displacement is known, the
184 compatible elastic distortion is obtained from its gradient and the total elastic distortion is
185 finally obtained by adding the incompatible part. The total, balanced, stress field $\boldsymbol{\sigma}$ is then
186 obtained by multiplication by the elastic stiffness tensor \mathbf{C} . Solving equations (6-8) for a given
187 distribution of Nye tensor provides a unique solution for the associated internal stress field [40].

188 Numerical solutions can be obtained from finite element approximations [41] and from spectral
189 methods [42]. The latter rely on the use of fast Fourier transform (FFT) algorithms [43] and
190 allow simulations of large three-dimensional volumes. Here, we employ an FFT spectral
191 approximation, based on the accelerated scheme [44] and using a special treatment for the
192 evaluation of the modified Green tensor in the Fourier space [45]. The reader is referred to

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3 193 reference [42] for details, whereby a very similar spectral algorithm was developed. We now
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5 194 describe the FFT simulation setup to model the dislocations observed in a thin foil by
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7 195 tomography and obtain their internal stress/strain fields. Figure 4 illustrates the main steps. We
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9 196 consider as an illustrative example a foil of dimension $960 \text{ nm} \times 960 \text{ nm}$ with a thickness of
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11 $\times 480 \text{ nm}$ shown in figure 4(a). A vertical screw dislocation line which crosses perpendicularly
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13 197 the two surfaces of the foil (threading dislocation) is inserted in the middle of the foil and shown
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15 198 by the solid black line. The FFT grid is here a cube made of $64 \times 64 \times 64$ voxels. The voxel
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17 199 size, i.e., the spatial resolution of FDM, is set to 15 nm in all simulations. Figure 4(b) shows
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19 200 the associated Nye tensor component α_{33} distributed on the FFT grid after the “Nye’zation”
20
21 201 procedure described above. Note that all dislocation densities are embedded inside the FFT
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23 202 volume. To create the two external free surfaces of the thin foils, where the stress field must
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25 203 satisfy zero-traction boundary conditions, we use a so-called gas phase, which is common in
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27 204 FFT simulations. Voxels with much lower elastic stiffness compose the gas phase. The
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29 205 interfaces between material voxels (with normal stiffness) and gas phase voxels are free
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31 206 surfaces, where the stress field satisfies zero-traction boundary conditions. An example of this
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33 207 gas phase method can be found in reference [42], where the stress field of an edge dislocation
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35 208 line near a pore was simulated and found to agree quantitatively very well with the existing
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37 209 analytical solution. In figure 4(b), one can see how the external free surfaces of the foil are
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39 210 created in the present FDM FFT simulation. Bright voxels compose the foil material, while dark
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41 211 voxels compose the so-called gas phase. More precisely, the elastic stiffness tensor \mathbf{C} of the
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43 212 olivine material will be attributed to each bright voxel, while a much lower stiffness (10^4 times
44
45 213 smaller) is attributed to the dark voxels. In doing so, the internal stresses will be affected near
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47 214 the two free surfaces to satisfy zero-traction boundary conditions. Figure 4(c) shows the internal
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49 215 shear stress field σ_{23} due to the dislocation line in the foil, obtained after convergence of the
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51 216 FDM-FFT numerical algorithm. One can see that it is affected by the external surfaces and
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3 218 tends to zero at them. Figure 4(d) also shows the elastic strain ϵ_{12} generated near and at the free
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5 219 surfaces to cancel the internal stresses σ_{13} and σ_{23} . A qualitative agreement is observed with
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7 220 elastic fields due to a threading screw dislocation in GaN, simulated by FDM with a finite
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9 221 element approximation [41]. Note finally that because the Nye dislocation density is
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11 222 numerically spread on FFT voxels, dislocation lines have an apparent core in the FDM
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13 223 simulations, with a size the order of the FFT voxel length (15nm), but it is to be interpreted as
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15 224 a numerical core, rather than a physical core. Note that the FDM can be applied to model
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17 225 dislocation cores [46], but it is not the scope of the present work. The spatial resolution is here
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19 226 15nm, much too large to properly describe real dislocation cores. The consequence of having
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21 227 numerical dislocation cores is that the internal stresses are smoothed and not well captured near
22
23 228 the physical dislocation cores, but far from the dislocation cores (a few nm) it is correctly
24
25 229 rendered. Short-term stress core corrections can be added [38, 39] and are important when
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27 230 dealing with small scale discrete dislocation dynamics, dislocation line tension effects and
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29 231 dislocation reactions. In the present work however, it is not critical and does not alter our
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31 232 findings, as we consider mostly straight dislocation lines separated by distances much larger
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33 233 than the physical core size of dislocations. Furthermore, the resolution (15nm) is already small
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35 234 such that large stresses between dislocation lines can already be predicted (see next section).
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42 Figure 4
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44 236 **4 Results and discussion**

45 237 **4.1 Dislocation microstructure**

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47 238 In order to take an overall look of the PoEM9 microstructure of dislocations, six tilt-series
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49 239 (obtained with the $22\bar{2}$ diffraction vector) were acquired at a low magnification, which
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51 240 represents an analyzed total area of approximately $30 \mu\text{m}^2$ [17], with an average angular range
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53 241 from -52° to 48° .
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3 242 Burgers vector indexations are performed using the thickness fringes technique [31] with the
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5 243 004 and $22\bar{2}$ diffraction vectors (Figures 1 and 2 respectively). They showed that the
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8 244 microstructures are only composed of dislocations with [001] Burgers vectors and
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10 245 overwhelmingly with straight screw dislocation characters. An example of dislocation
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12 246 microstructure obtained in WBDF conditions with the 004 diffraction vector is shown on
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14 247 Figure 1. The electron tomography shows a majority of {110} and (100) glide planes, few
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16 248 cross-slip configurations [17] and colinear interactions [17]. The reconstructed volumes also
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19 249 provide access to (\mathbf{u}, \mathbf{b}) doublets for each dislocation segments, to calculate the Nye tensor,
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22 250 and to simulate the continuous distribution of stress field.
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24 251 Two domains of specimen PoEM9 are studied in detail: zone 1 on Figures 5a-d and zone 2 on
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26 252 Figures 5e-h. A tilt-series composed of 51 micrographs (acquired every 2° with a tilt angle
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28 253 ranging from -48° to $+52^\circ$) and a second tilt-series of 54 micrographs (acquired every 2° with
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30 254 a tilt angle ranging from -56° to $+50^\circ$) were obtained in WBDF conditions with the $22\bar{2}$
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32 255 diffraction vector (zones 1 and 2 respectively). Micrographs from Figures 5a and 5e come from
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34 256 these two tilt-series for a projection angle of 0° . Electron tomography reconstructions enable
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36 257 the characterization of the (110), $(1\bar{1}0)$ and (100) slip systems which are identified by their
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38 258 colours: white, light grey and black respectively as in Mussi *et al.*, 2015 [17]. Neither the slip
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40 259 systems of the pure screw straight dislocations (coral pink-coloured), nor the habit plane of the
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42 260 red sessile dislocation segment in Figure 5f, can be indexed. Then, the reconstructed volumes
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44 261 are projected along the [001] direction in order to orientate the pure screw dislocations in edge-
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46 262 on position (Figures 5c and 5g) and thereafter, to provide optimal viewing conditions to study
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48 263 the continuous stress distributions.
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265 **4.2 Simulation results**

266 The dislocation internal stresses in the thin foil are rotated in the olivine crystal reference frame.
 267 The x_1 , x_2 and x_3 directions are now aligned with the a , b and c directions of the olivine
 268 orthorhombic unit cell, respectively. In addition to this internal stress, we add the macroscopic
 269 stress applied experimentally during the compression test (754 MPa) also rotated in the crystal
 270 reference frame. This is possible since we know the angle between the normal of the thin foil
 271 $[17 \bar{5} 12]$ and the compression axis $[502]$ (approximately 30°). The total stress field at every
 272 voxel in the thin foil is then $\sigma_{ij} = \sigma_{ij}^{int} + \Sigma_{ij}$, where σ_{ij}^{int} is the local internal stress field due to
 273 dislocations and Σ_{ij} is the macroscopic stress. It is then possible to project the total stress tensor
 274 at every voxel on different slip systems using the associated Schmid tensor expressed in the
 275 olivine orthorhombic reference frame. In the following, we consider three slip systems, labelled
 276 1, 2 and 3: $[001](100)$, $[001](110)$ and $[001](1\bar{1}0)$ respectively. The resolved shear stress
 277 (RSS) for the three systems are denoted by RSS1, RSS2 and RSS3 in the following. They are
 278 equal to:

$$\begin{aligned}
 279 \text{RSS1} &= \tau^{[001](100)} = \sigma_{31}, \\
 280 \text{RSS2} &= \tau^{[001](110)} = \frac{b}{\sqrt{a^2+b^2}} \sigma_{31} + \frac{a}{\sqrt{a^2+b^2}} \sigma_{32} \approx 90.63 \% \sigma_{31} + 42.26 \% \sigma_{32}, \\
 281 \text{RSS3} &= \tau^{[001](1\bar{1}0)} = \frac{b}{\sqrt{a^2+b^2}} \sigma_{31} - \frac{a}{\sqrt{a^2+b^2}} \sigma_{32} \approx 90.63 \% \sigma_{31} - 42.26 \% \sigma_{32}. \quad (9)
 \end{aligned}$$

282 with a and b the directions of the olivine orthorhombic unit cell.

283 The macroscopic stress $\Sigma_{31} = 10ac\sigma / [(5a)^2 + (2c)^2] \approx 302.8$ MPa (with a and c the directions
 284 of the olivine orthorhombic unit cell and σ the applied stress) while $\Sigma_{32}=0$. Figures 5 and 6
 285 show the spatial distribution of the three resolved shear stresses on slices in the thin foil, for the
 286 two thin foil series. Dislocation lines are shown in figure 5. They are removed in Figure 6 for a
 287 better comparison of RSS distributions in different slip systems. RSS1, RSS2 and RSS3 fields
 288 look similar, local rotations of RSS patterns (lobes) near dislocations can be seen between the
 289 three RSS maps. The distribution of the internal elastic energy density $1/2 \varepsilon_{ij}^e C_{ijkl} \varepsilon_{kl}^e$ is also

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3 290 shown in Figure 6. It shows elastic interactions between dislocations (interacting elastic fields
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5 291 between dislocations). Note that the calculation of the mean internal elastic energy within the
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7 292 thin foil volume give 51.6 kJ/m³ for series 1 and 52.2 kJ/m³ for series 5. The reader is further
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9 293 referred to animations in the supplementary materials, where moving slices parallel to slip
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11 294 planes allow for a better visualization of the long-range elastic interactions between dislocation
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13 295 lines within the thin foils. Histograms of the RSS distributions in the thin foils are provided in
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15 296 Figure 7(a, b) showing a significant heterogeneity of the stress field within the thin foils. One
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17 297 can note that the mean value of RSS1 (302.8 MPa) is slightly larger than that of RSS2 and RSS3
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19 298 (274.4 MPa). This is because the sample was oriented to activate system 1 preferentially. Strong
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21 299 variations in the stress values can be seen, typically ± 400 MPa around the mean values.
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23 300 Regions with local stresses reaching 1 GPa are also observed, particularly nearby dislocation
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25 301 dipoles. Finally, Figure 7(c) shows cumulative distribution functions (CDF) for RSS1, RSS2,
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27 302 RSS3, for the two thin foil series, with RSS values extracted only at the dislocation lines
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29 303 (instead of taking values at all voxels in the thin foils). In doing so, we only consider dislocation
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31 304 driving stresses in the single crystal. The CDF are centred about zero. Broad distributions, with
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33 305 again variations up to ± 400 MPa, are clearly evidenced in the CDF for the three slip systems
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35 306 and the two thin foils, and they look rather similar. One clear feature in all these maps and
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37 307 distributions is thus the presence of long-range elastic interactions between dislocations and
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39 308 significant local variations of the RSS values in the three systems considered due to the
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41 309 complexity of the three-dimensional stress state.
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51 311 **4.2 Discussion**

52 312 The present coupled electron tomography/FDM analysis allows obtaining for the first time a
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54 313 detailed, three-dimensional, physical/mechanical description of dislocation networks in the
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56 314 studied thin foils. A physical description of dislocations (Burgers vectors, 3D arrangement of
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3 315 dislocation lines, junctions, dipoles, networks) is complemented by a mechanical description
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5 316 (strains, rotations, stresses, energy). In the present study, we specifically discuss two points, (1)
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7 317 the possible activation of different slip systems in olivine deformed at low temperature due to
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9 318 the complexity of the three-dimensional stress state, and (2), the history of dislocation dynamics
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11 319 and the associated build-up of polarized internal stresses and kinematic hardening in single
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13 320 crystals at the nanometre scale.
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19 322 (1) Activation of slip systems: The POEM-9 specimen, from which thin foils have been
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21 323 extracted and analysed here, was compressed along the [502] direction, to preferentially
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23 324 activate the [001](100) slip system (system 1 and RSS1). However, an uncertainty between
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25 325 the possible activated slip systems was mentioned [29]. Another system, [001](110) was also
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27 326 proposed as a possible system. A third system could also be activated, [001](1 $\bar{1}$ 0). They
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29 327 correspond to system 2 and system 3 in the present study. Our distributions of RSS values in
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31 328 the thin foil indeed suggest that system 1 is only slightly favoured as compared to systems 2
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33 329 and 3, because the mean value of RSS1 is only slightly larger (difference of less than 30 MPa).
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35 330 However, in a recent work [17], systems 2 and 3 are observed to be easier to activate (lower
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37 331 CRSS) than system 1, system 1 being the second easiest system to activate after system 2 and
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39 332 3. As such it is not easy to say which one of system 1 or systems (2, 3) was preferentially
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41 333 activated in our studied sample. The distributions of the RSS values extracted only at
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43 334 dislocation lines are certainly more speaking. Indeed, the distributions show very similar
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45 335 features for all series and slip systems. More precisely, they are all broad and show significant
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47 336 local variations, ± 400 MPa. As such, the slight difference of the mean RSS between system 1
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49 337 and systems (2, 3), less than 30 MPa, becomes negligible as compared to possible large local
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51 338 values of RSS. From this, we can conclude that, although POEM-9 was compressed in a way
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339 to favor slip system 1, the three systems can all be activated, because of the strong heterogeneity
340 of the internal stress field.

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342 (2) Internal stresses and kinematic hardening: The simulated fields of RSS show a significant
343 heterogeneity. Recently, an experimental work on deformed olivine single crystals and
344 polycrystals using High-Resolution-EBSD (HR-EBSD) revealed a pronounced heterogeneity
345 in the in-plane elastic shear strains and shear stresses at the surface [47, 48]. Variations up to
346 ± 1 GPa around the mean value were reported in both polycrystals and single crystals (at low
347 temperatures) at the micron scale. Such polarization and patterning of internal, intragranular
348 and intergranular stresses, can be related to the build-up of geometrically necessary dislocation
349 densities. Strong heterogeneity of internal stresses is to be associated with kinematic hardening.
350 In the present study, we can look at the heterogeneity of internal stresses down to the scale of
351 typical HR-EBSD map pixels, *i.e.*, at the nanometre scale. Our results also reveal a significant
352 heterogeneity of internal stresses at the nanometre scale in a single crystal containing
353 dislocation networks. Distributions of RSS show broad profiles, with variations up to ± 400
354 MPa around the mean value. Interestingly, the histograms show a few negative RSS values.
355 The results thus suggest that kinematic hardening has been built-up and is a signature of the
356 past dynamics of dislocations observed.

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358 The application of our electron tomography/FDM method brings valuable insights regarding
359 the complexity of plastic flow in olivine. In a simple single crystal subjected to simple
360 compression loading, it shows that plastic anisotropy and activation of dislocation slip systems
361 can be significantly altered by the large fluctuations of the local mechanical fields. Furthermore,
362 it evidences the built-up of kinematic hardening. This plastic behaviour observed at the single
363 crystal, nanometre scale, certainly has a strong impact on the rheology of olivine polycrystals

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3 364 within the Earth's mantle. More generally, our proposed analysis method can also be employed
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5 365 to investigate other mechanisms and defects typically investigated in the TEM for various
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7 366 crystals, such as subgrain boundaries, dislocation networks, dislocation grain boundary
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9 367 interactions, dislocation reactions, interactions between dislocations and pores/precipitates etc.
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11 368 Furthermore, as shown in Figure 8, the method also provides elastic fields in the vicinity of and
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13 369 at free surfaces, which allows making links between elastic fields measurements using
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15 370 experimental techniques such as precession electron diffraction [49], HR-EBSD (high-
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17 371 resolution electron backscatter diffraction), HR-TKD (high-resolution transmission Kikuchi
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19 372 diffraction) [41], ACOM (automated crystal orientation mapping) ASTAR[®] [50] and defects
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21 373 inside the material. It must be emphasized that, in addition to provide 3D anisotropic elastic
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23 374 fields, our approach allows a spatial resolution of 15 nm in routine use, extendable down to 5
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25 375 nm. The method can also be used to assess the accuracy of HR-TKD, or ACOM/ASTAR
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27 376 methods, when measuring the elastic fields around dislocation structures.
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34 35 378 **5 Conclusion**

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37 379 In this contribution, we propose an original experimental and theoretical characterization
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39 380 method for studying dislocation networks in crystalline materials. The method combines
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41 381 dislocation electron tomography, which provides a three-dimensional characterization of
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43 382 dislocation lines and Burgers vectors, to a field dislocation mechanics model, which provides
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45 383 the associated internal mechanical fields. To demonstrate the potential of our method, we have
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47 384 considered single crystal thin foils extracted from a compressed olivine single crystal. Our
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49 385 results have revealed significant local fluctuations in the local mechanical fields, particularly
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51 386 the resolved shear stresses in different slip systems, which certainly play a role in the strong
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53 387 anisotropy of olivine and the activation of different dislocation slip systems. Our first results
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55 388 have also evidenced the built-up of kinematic hardening at the nanometre scale in a single
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389 crystal. Our method has a considerable potential for studying various types of dislocation
390 microstructures and plastic deformation mechanisms in different crystals

391

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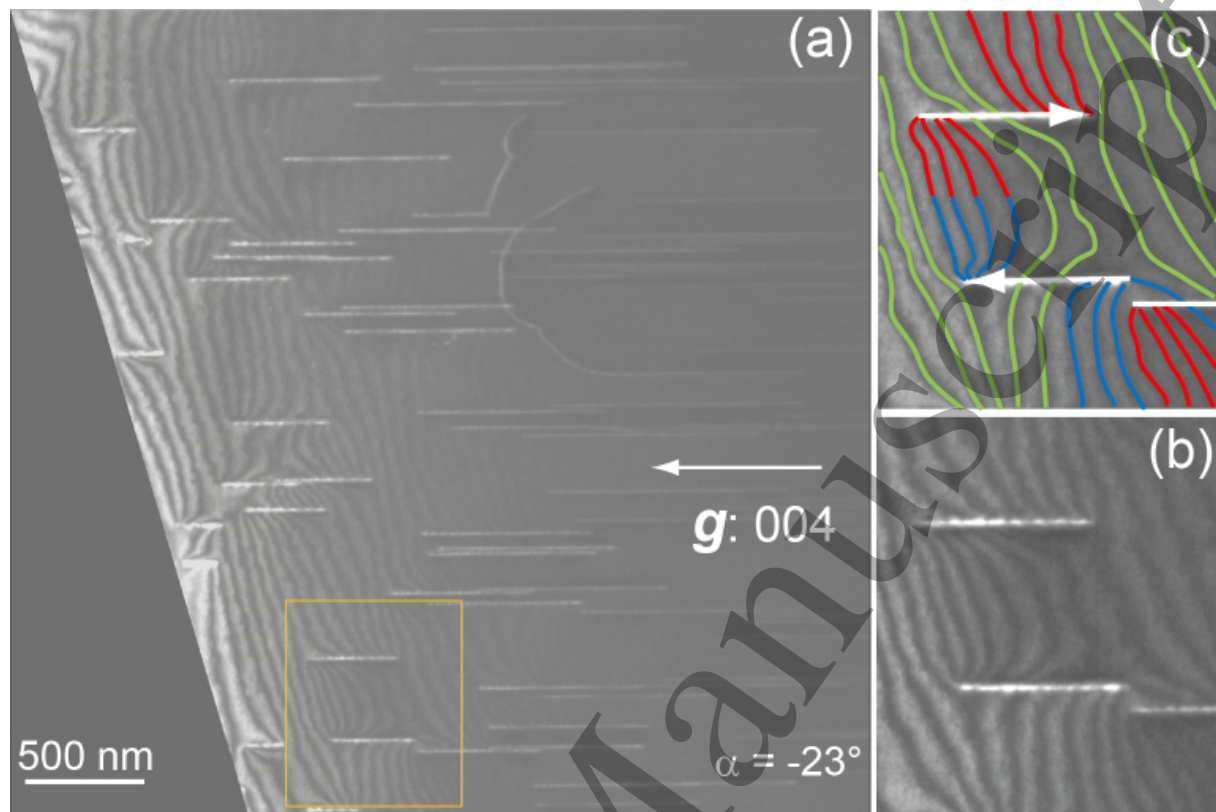
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551 **Figures**

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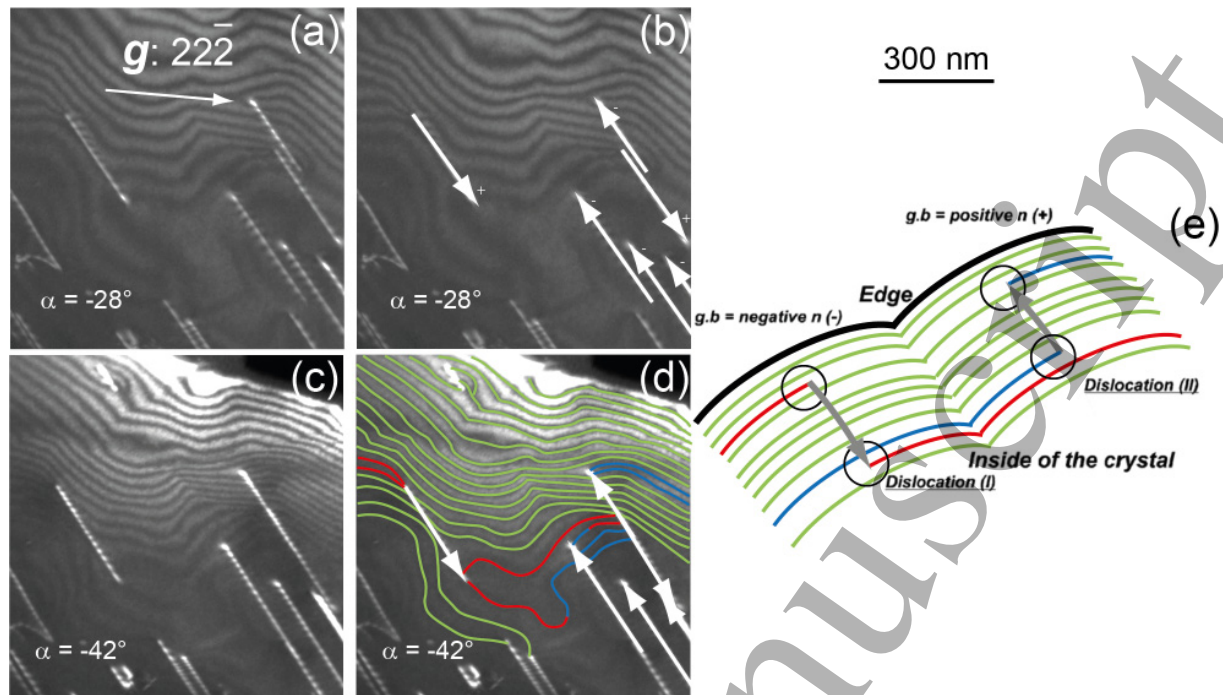


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554 **Figure 1:** WBDF micrograph obtained with the 004 diffraction vector: (a) Global micrograph
 555 where thickness fringes are clearly seen; (b) enlargement of a dislocation dipole pointed out by
 556 an orange rectangle on Figure 1(a) ; (c) enlargement of the dipole where the unterminated
 557 thickness fringes are coloured in green, the terminated thickness fringes are coloured in red at
 558 the extremities of the dislocations represented with arrows oriented on the opposite direction of
 559 the wedge, and the terminated thickness fringes are coloured in blue at the extremities of the
 560 dislocation represented with an arrow oriented on the direction of the wedge (the directions of
 561 the arrows are linked with the signs of the Burgers vectors).

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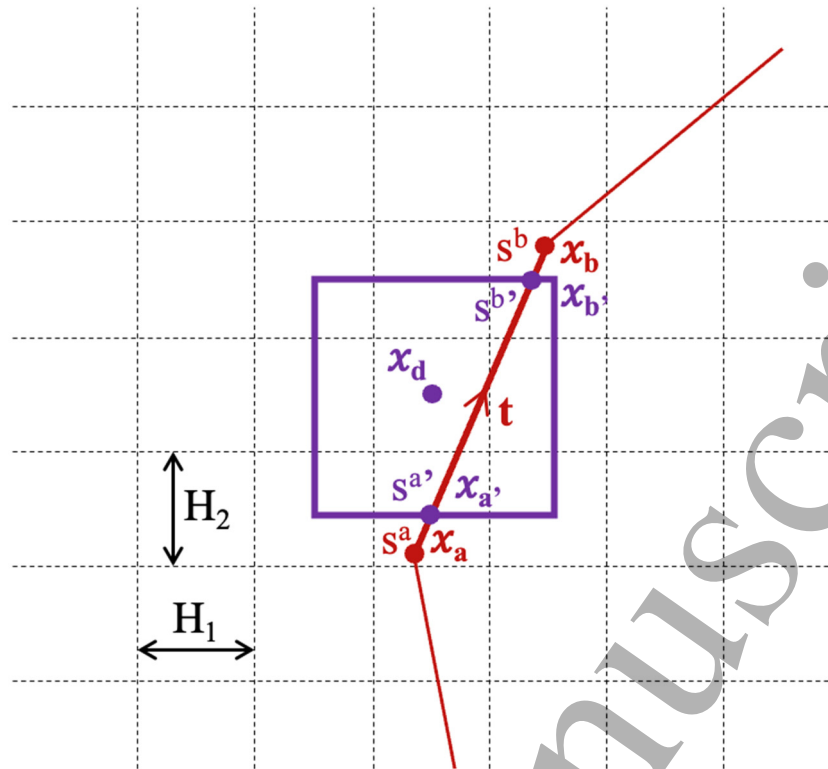
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564 **Figure 2:** Association of the intensity of the dislocation extremities with the sign of the Burgers
 565 vector: (a) Raw micrograph in WBDF conditions, obtained with the $22\bar{2}$ diffraction vector (with
 566 a projection angle of -28°), where an intensity asymmetry of dislocation extremities is clearly
 567 seen; (b) arrows added on the raw micrograph of Figure 2(a) = the arrow is oriented in the
 568 direction of the intense extremity of the dislocation; (c) raw micrograph of the same domain
 569 with a projection angle of -42° ; (d) arrows and thickness fringes added and redrawn on the raw
 570 micrograph of Figure 2(c) where the thickness fringes are coloured in green when they are not
 571 terminated on the extremities of dislocations, in red at the extremities of the dislocations
 572 represented with arrows oriented on the opposite direction of the wedge, and in blue at the
 573 extremities of the dislocation represented with an arrow oriented on the direction of the wedge;
 574 (e) cartoon extracted from Miyajima & Walte [32], where the method to get access to the $\mathbf{g} \cdot \mathbf{b}$
 575 product is described, taking into account the signs of the Burgers vectors, following Ishida *et*
 576 *al.* [31], with the same color code and arrow direction code as Figure 2(d). A link between the
 577 intensity of the dislocation extremities and the sign of the Burgers vector is noticeably evident.

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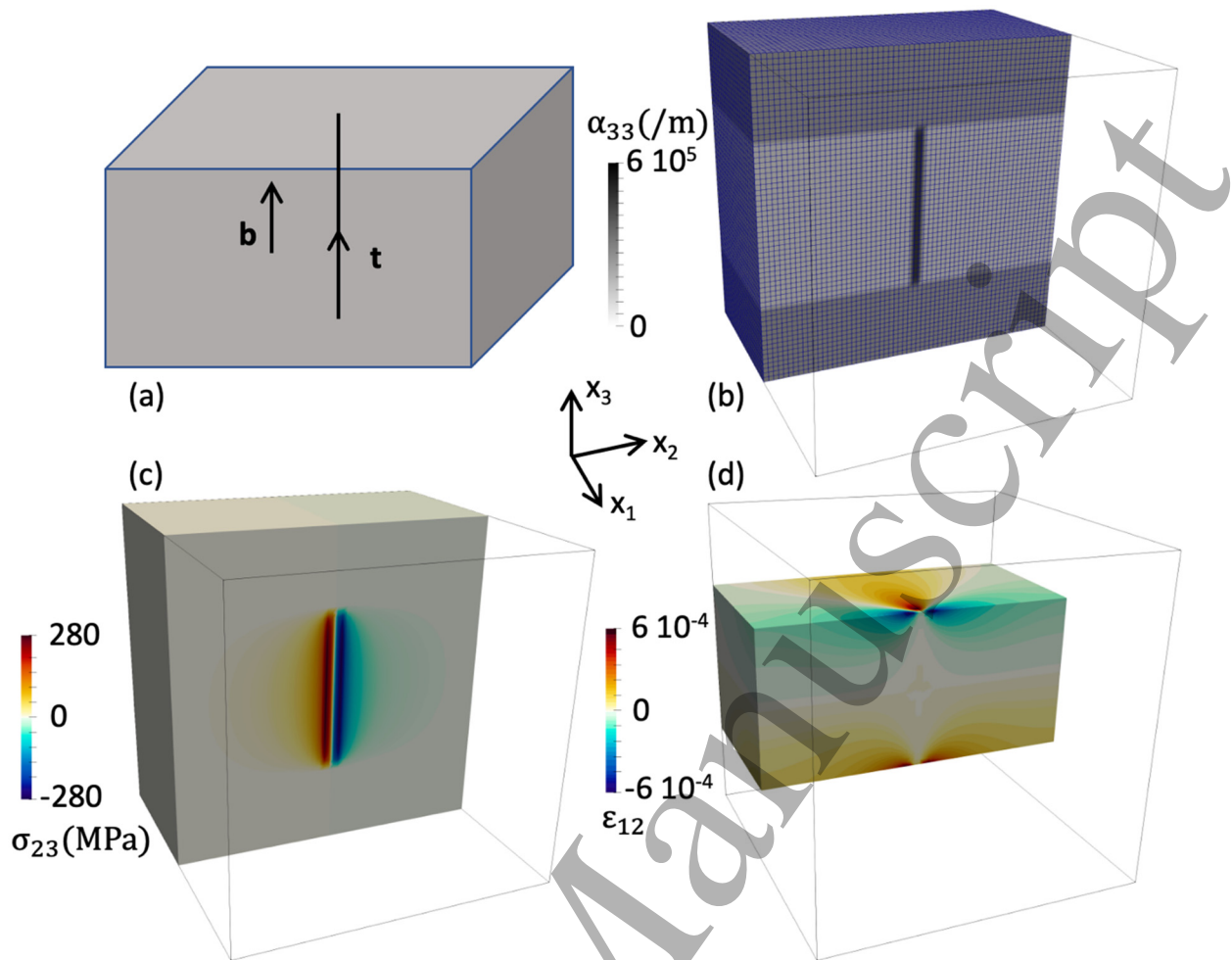


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579 **Figure 3:** Illustration of the method used to assign a Nye dislocation density at a voxel x_d due
 580 to a dislocation segment. See text for details.

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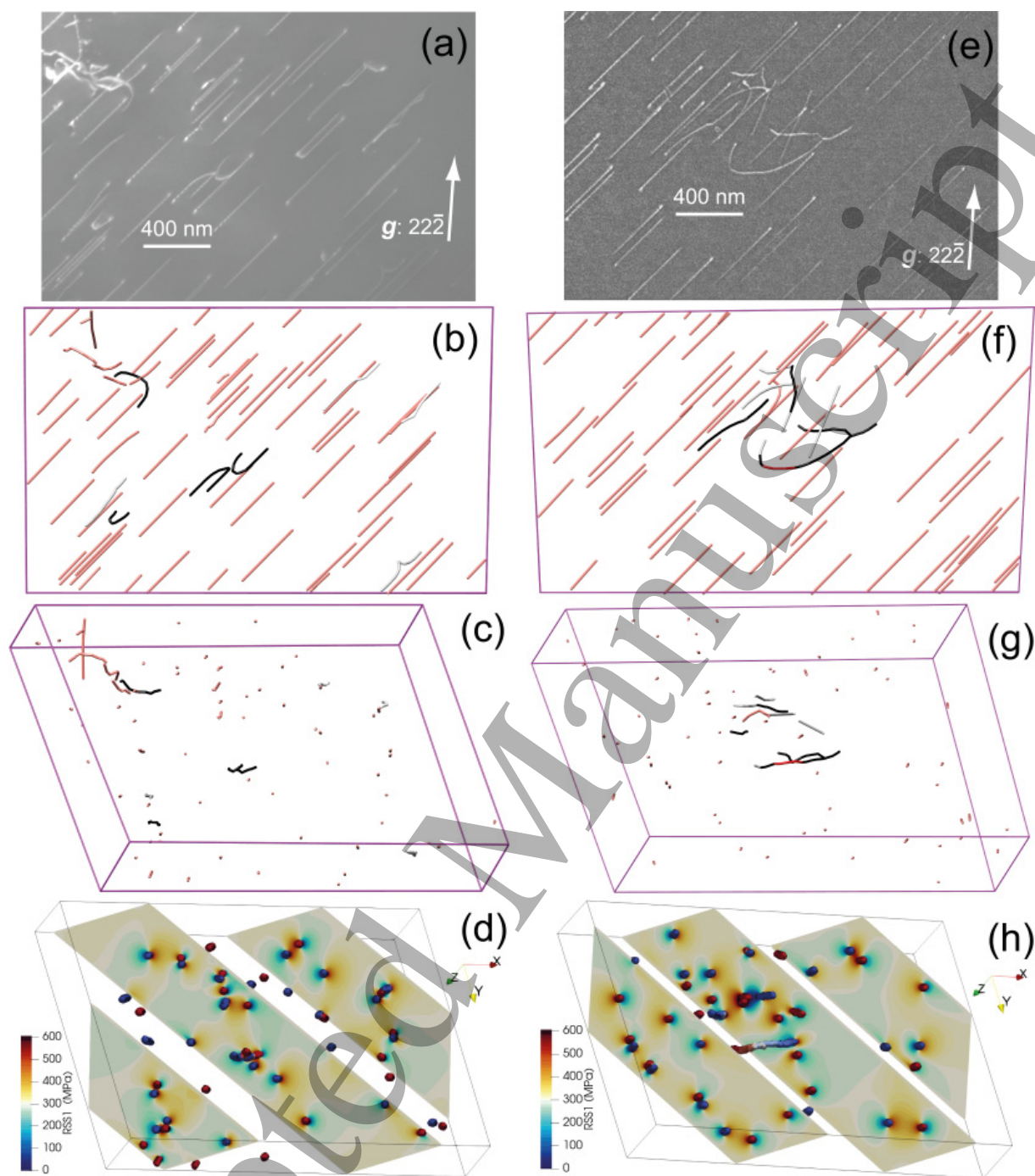


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583 **Figure 4:** Numerical strategy to model the stress field of discrete dislocation lines observed by
 584 tomography in a thin foil. (a) Sketch of a thin foil containing one vertical threading screw
 585 dislocation line (black solid line, the line and Burgers vectors are also indicated). (b)
 586 Corresponding Nye tensor screw component α_{33} on the 3D FFT grid (clip). Bright voxels
 587 belong to the thin foil while dark voxels correspond to the gas phase. (c) Internal shear stress
 588 field σ_{23} . (d) Elastic strain field ϵ_{12} generated near and at the free surfaces of the thin foil to
 589 cancel the internal stresses σ_{13} and σ_{23} at the free surfaces. The coordinate system used for
 590 figures (b, c, d) is shown at the centre of the figure.

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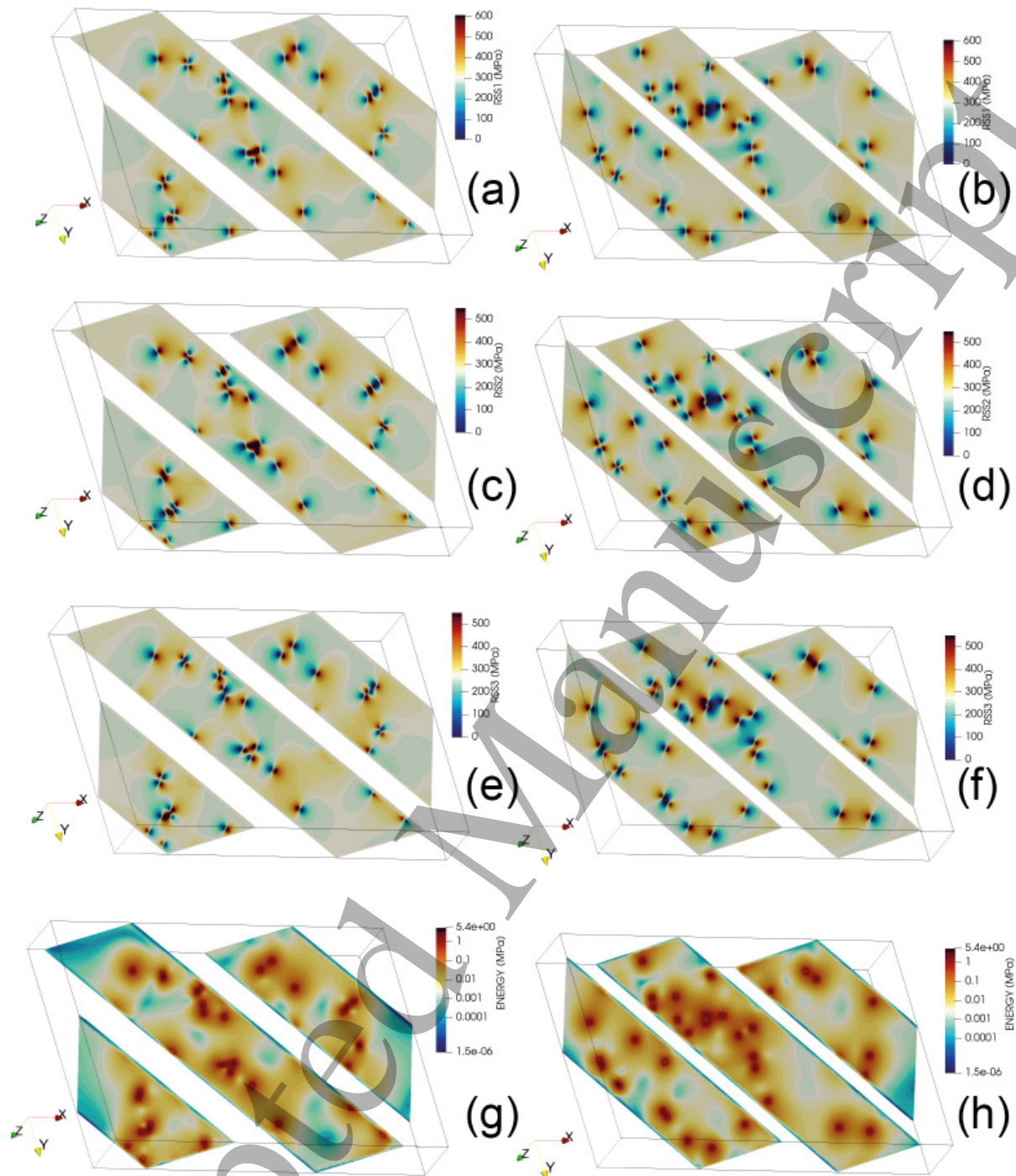


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593 **Figure 5:** Link between TED and FDM: (a) Raw micrograph in WBDF conditions, obtained
 594 with the $22\bar{2}$ diffraction vector (with a projection angle of 0°) of zone 1; (b) reconstructed
 595 volume of the same domain with the same projected angle (the (110), $(\bar{1}10)$ and (100) slip
 596 systems are respectively colored in white, light grey and black as in [17]); (c) reconstructed
 597 volume of the same domain along the [001] direction; (d) Spatial distributions (3 slices) of
 598 RSS1 in series 1 obtained along the same orientation as Figure 5(c) located in the interaction

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3 599 between the two black dislocations in the middle of the micrograph in Figures 5(a-b)
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5 600 (Dislocation lines are also shown and coloured w.r.t. their sign.); (e) raw micrograph in WBDF
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7 601 conditions, obtained with the $22\bar{2}$ diffraction vector (with a projection angle of 0°) of zone 2;
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9 602 (f) reconstructed volume of the same domain with the same projected angle (the $(1\bar{1}0)$ and
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11 603 (100) slip systems are respectively colored in light grey and black); (g) reconstructed volume
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13 604 of the same domain along the $[001]$ direction; (h) Spatial distributions (3 slices) of RSS1 in
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15 605 series 5 obtained along the same orientation as Figure 5(g) located in the red dislocation
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17 606 segment (Dislocation lines are also shown and coloured with reference to their sign).
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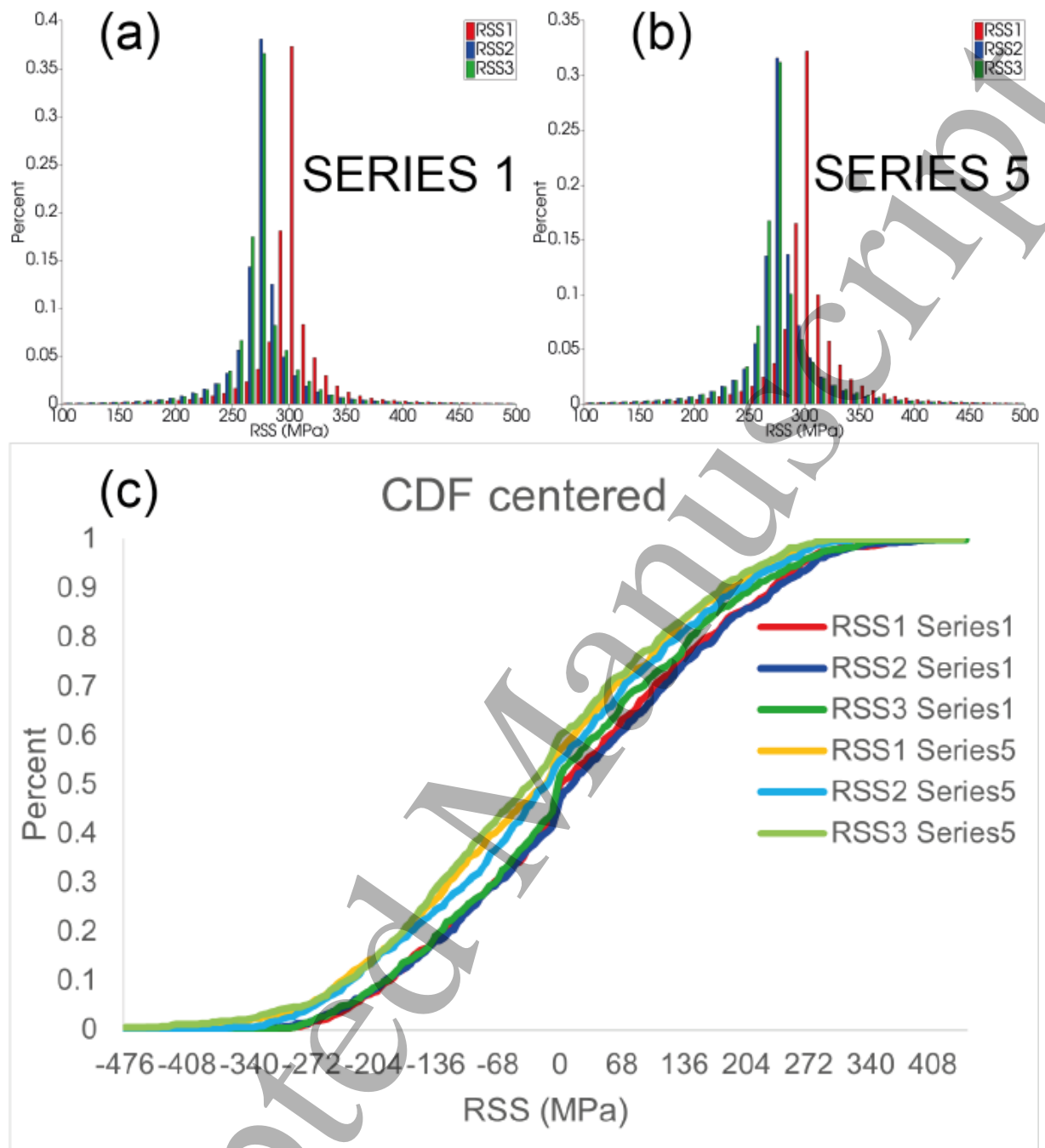
609 **Figure 6:** Spatial distributions (3 slices) of RSS1 (a, b), RSS2 (c, d), RSS3 (e, f) and elastic

610 energy density (g, h) for series 1 (left column) and series 5 (right column).

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614 **Figure 7:** Distributions of RSS1, RSS2 and RSS3 for series 1 (zone 1) and series 5 (zone 2).

615 (a, b): All RSS values in the thin foils are used. (c): Only RSS values at dislocation lines are

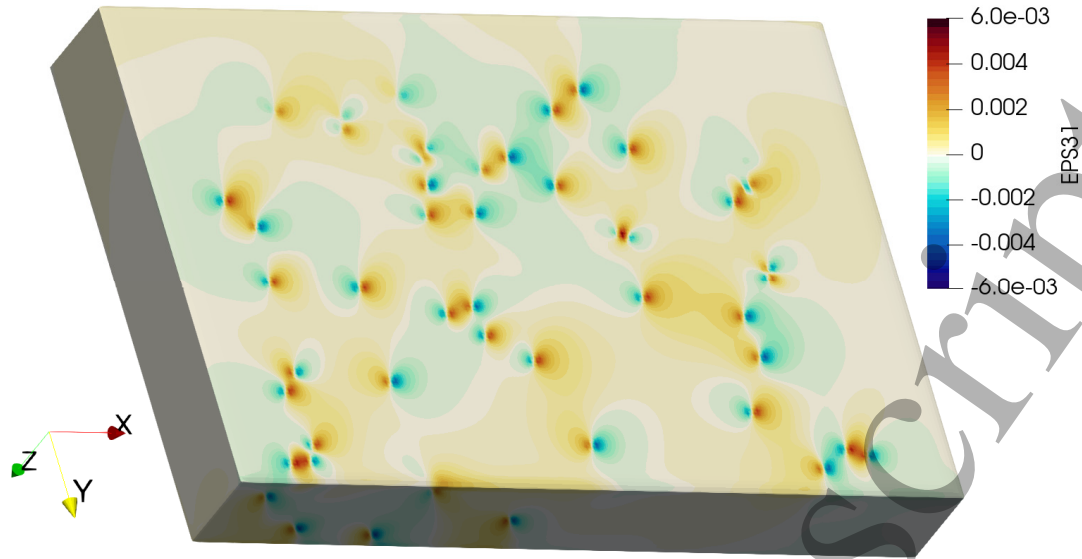
616 used. All cumulative distribution functions (CDF) for the three slip systems and the two series

617 are superimposed on the same figure.

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620 **Figure 8:** Elastic strain ϵ_{31} at the surface of the thin foil for series 1.

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