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# 1           **Orientation and deformation conditions dependence of** 2           **dislocation substructures in cold deformed pure tantalum**

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10

## 11    **Abstract**

12    The evolution of dislocation arrangement during deformation can lead to the development of  
13    dislocation substructures which have direct consequences on the subsequent microstructural  
14    evolutions. Dislocation densities and substructures of various deformed microstructures of  
15    pure tantalum are analyzed in order to study the influence of deformation conditions. Electron  
16    BackScatter Diffraction (EBSD) and Electron Channeling Contrast Imaging (ECCI) are used  
17    in order to characterize the development of dislocation substructures. Investigation at grain  
18    scale shows the orientation dependence of the deformed state with the highest dislocation  
19    content for  $\gamma$ -fiber grains. This fiber appears to be quite stable in compression but less in  
20    rolling. Investigation at dislocation substructure scale highlights the influence of orientation  
21    on dislocation substructure forming. Developed substructures are present in the interior of  $\gamma$ -  
22    fiber grains. The evolution of the disorientation and size of substructures with strain depends  
23    greatly on the initial fraction of  $\gamma$ -fiber in the microstructure. Models describing these  
24    evolutions are proposed for  $\gamma$ -fiber grains. On the basis of the present analysis it is  
25    concluded that dislocation density parameter does not allow transcribing the differences of  
26    substructural development.

27

## 28    **Keywords**

29    Tantalum; Cold deformation; Dislocation; Substructure; Crystallographic orientation; EBSD

30

## 31 1. Introduction

32 During cold deformation of a metallic alloy, a small amount of work ( $\approx 1\%$ ) is stored in the  
33 material and is correlated to generated dislocations [1]. This stored energy is the driving  
34 force for several thermally activated physical mechanisms during subsequent annealing such  
35 as recovery and recrystallization. These mechanisms determine the mechanical properties of  
36 the final part [2,3] therefore it is crucial to analyze and quantify the stored energy distribution  
37 in order to model recrystallization.

38 Tantalum is a bcc high density ( $16654 \text{ kg.m}^{-3}$ ) refractory metal ( $T_m = 2996^\circ\text{C}$ ) [4]. Thanks to  
39 its high ductility at room temperature, it can be employed to cold form parts with complex  
40 geometries with minimum damage risks. Pure tantalum and tantalum alloys have been used  
41 in many fields such as medical field, electronics or chemical industries for instance [5,6].

42 For body centered cubic (bcc) materials, three slip systems can be activated:  $\{110\} \langle 111 \rangle$ ,  
43  $\{112\} \langle 111 \rangle$  and  $\{123\} \langle 111 \rangle$ , all three having the same slip direction giving dislocations the  
44 possibility to move from one slip plane to another [7]. The activation of those slip systems  
45 depends on deformation temperature, strain rate, chemical composition and crystal  
46 orientation [1,7]. For Ta, it was generally observed that even though slip activity is mainly on  
47  $\{110\}$  planes, it is also present on plane with maximal resolved shear stress [8]. Moreover,  
48 stacking faults have been rarely observed experimentally for bcc materials and the  
49 computation of energy with voluntarily created stacking faults has confirmed that stable faults  
50 are unlikely to exist. Therefore, the stacking fault energies are often estimated only  
51 theoretically with some assumptions [9,10]. The ease of cross slip suggests a very similar  
52 dislocation behavior to high stacking faults face centered cubic (fcc) materials [7] such as  
53 aluminum. With cross slip ability, dislocations tend to form well-organized substructures  
54 during plastic deformation.

55 Due to the micrometer range of substructures, Transmission Electron Microscopy (TEM) is  
56 mostly used to characterize microstructures. Size and shape of substructures were mainly  
57 studied in deformed fcc metals such as Al [11–14], Ni [15,16], Cu [14,17] or austenitic 304L  
58 stainless steel [15]. Results have shown the decrease of substructure size with increasing  
59 strain. The work of Gil Sevillano et al. [18] revealed that a wide range of materials displayed  
60 a similar behavior including bcc materials and experiments on cold worked Nb [19] and iron  
61 [20] have confirmed this observation. The evolution of the substructure disorientation with  
62 deformation has also been investigated but to a lesser extent. Some studies on this topic  
63 have been carried out on bcc materials such as iron [18,20] but most of the investigations  
64 have been realized once again on fcc materials in particular on Al [11–14,21,22]. It has been

65 found that generally the substructure disorientation increased with strain. An attempt to  
66 determine a universal behavior of dislocation substructures has been done. For that purpose,  
67 substructures boundaries, also named subboundaries, were classified in two different types  
68 [23]: Incidental Dislocation Boundaries (IDBs) and Geometrically Necessary Boundaries  
69 (GNBs). Pantleon and Hansen [24] demonstrated that the disorientation distribution of  
70 dislocation boundaries can be described with a Rayleigh function of IDBs and GNBs.  
71 Pantleon [25] demonstrated that the saturation of boundaries disorientations frequently  
72 observed is an artefact due to both the threshold disorientation angle used to differentiate  
73 dislocations boundaries from grain boundaries and the superposition of IDBs and GNBs.

74 Electron Back Scatter Diffraction (EBSD) technique has emerged in the last decades and  
75 became a powerful tool to characterize microstructures. Although the limiting factor of EBSD  
76 concerning its angular resolution ( $\approx 0.5^\circ$ ), this technique provides data of much better  
77 statistical relevance than TEM data thanks to bigger analyzed surfaces. Therefore, a  
78 quantitative characterization of dislocation substructures on larger scales is achievable.  
79 Similarly to TEM studies, pure Al and Al alloys deformed microstructures have been  
80 intensively characterized with EBSD technique [26–29]. The observed evolutions of  
81 substructure size and disorientation were consistent with TEM observations. Less  
82 investigations on bcc metals such as pure Nb [30] or pure Ta [31,32] have been generally  
83 carried out but have also reported similar results.

84 The influence of crystallographic orientation on grain deformation has been also a topic  
85 extensively under investigation. In order to have a better understanding of the  
86 recrystallization texture, Dillamore et al. [33] observed with TEM technique that substructure  
87 size and disorientation were orientation dependent for cold rolled iron. Substructure size  
88 decreased and disorientation increased as the rolling plane of the grains rotated from {001}  
89 to {110} around the rolling direction  $\langle 110 \rangle$ . Similar orientation dependence for the stored  
90 energy has been found on rolled steel using a X-ray line broadening method [34] and more  
91 recently on cold worked high purity iron using a high resolution X-ray method [35].  
92 Orientation dependence of substructure development has been also observed for fcc metals  
93 such as for rolled pure Al [36] characterized with TEM technique where the substructures  
94 properties evolved differently depending on the considered rolling texture component.  
95 Complementary information on the deformation substructures have been found with EBSD  
96 investigations for instance on Al alloy [28] where it has been shown that the crystallographic  
97 orientation influenced mainly the substructure disorientation but not much their size. For the  
98 specific case of pure Ta, this influence has been also studied and the work of Sandim et al.  
99 [37] on cold rolled coarse-grained high-purity Ta has clearly illustrated it with two grains with  
100 different substructure development: one grain subdivided into regions with a large range of

101 disorientations whereas the other one deformed in a more stable way with low misoriented  
102 subboundaries. Moreover, EBSD investigations on cold rolled pure Ta [38–40] have shown  
103 that more generally grains with the direction  $\langle 111 \rangle$  parallel to the normal direction of the  
104 sheet contained more substructures and had a higher stored energy, quantified with the  
105 quality of EBSD patterns, than grains with the direction  $\langle 100 \rangle$  parallel to the normal  
106 direction.

107 Although these previous studies have indicated that individual grains behaved quite  
108 differently during the deformation because of crystallographic orientation, existing literature  
109 has not reported quantitative characterization of the evolution of the spatial distribution of  
110 substructures with strain for pure Ta. Indeed, not only the disorientation of substructures is of  
111 interest but also the spatial organization of the substructures has to be studied because it  
112 can impact the subsequent annealing mechanisms. For instance, Yu et al. [41] have found  
113 that the triple junction formed by the substructures in cold rolled Al may act on recovery and  
114 on recrystallization. The triple junction motion during recovery allowed a structural  
115 coarsening and the formed equiaxed structures might represent possible nuclei for  
116 recrystallization.

117 In the current investigation, the evolution of dislocation substructures during room  
118 temperature plastic deformation is investigated in pure Ta. Samples were deformed by  
119 uniaxial compression and rolling. An investigation of the orientation dependence of  
120 dislocation content and dislocation substructure development is performed. Two scales of  
121 observation with the EBSD technique are used in order to characterize the deformed  
122 microstructures. Firstly, at grain scale to examine the evolution of the texture and of the  
123 dislocation density with increasing strain. Secondly, at dislocation substructure scale to  
124 observe and quantify with a lower resolution the spatial distribution and development of  
125 substructures. Correlations between macroscopic and microscopic observations are then  
126 discussed. Parameters to quantify substructure development are defined and models for its  
127 evolutions with strain are proposed.

128

## 129 **2. Material preparation and SEM characterization**

130 For microstructural analysis, surfaces were prepared by mechanical polishing up to 4000 grit  
131 SiC paper. Several papers were used for each step to ensure the removal of the hardened  
132 layer of the previous step and not simply erase the previous scratches. A chemical-  
133 mechanical polishing was then performed with a colloidal silica suspension with an average

134 particle diameter of 20 nm. The samples were finally etched with a solution of 25% HF and  
135 75% HNO<sub>3</sub> for several seconds.

136 EBSD scans were performed on a Carl Zeiss Supra40 Field Emission Gun Scanning  
137 Electron Microscope (FEG-SEM) operated under 20 kV with a Bruker Quantax system  
138 comprising a FlashHR EBSD detector and the Esprit 2.1 EBSD software package. EBSD  
139 maps were acquired at the samples mid-thickness. For intergranular observations, an  
140 acquisition step size of 1.20 μm was used over a rectangular area of 1.20 mm × 0.90 mm.  
141 This measurement step size was chosen to achieve a compromise between the  
142 measurement area and the spatial resolution in order to have statistically relevant data. For  
143 intragranular observations, EBSD maps with an acquisition step size of 90 nm were  
144 performed in the interior of the grains. This measurement step size allows the distinction of  
145 close dislocation subboundaries even in the most deformed cases. For EBSD data  
146 treatments, MTEX toolbox was used [42]. Electron Contrast Channeling Imaging (ECI)  
147 micrographs under 30 kV with a backscatter electron detector were also performed at this  
148 scale for a direct observation of dislocation substructures.

149

### 150 **3. Initial state**

151 The as-received material is a sheet of 12 mm thickness of pure tantalum (> 99.99% wt) that  
152 was subject to cold rolling then to a recrystallization heat treatment. The initial fully  
153 recrystallized microstructure exhibits heterogeneities between the center and the quarter  
154 layer (3 mm from the center) of the sheet as illustrated in Fig. 1. In order to study texture  
155 evolutions, grains were classified into the following three groups based on their average  
156 orientations: the  $\theta$ -fiber grains with  $\langle 100 \rangle$  parallel to the normal direction of the sheet (ND)<sup>1</sup>,  
157 the  $\gamma$ -fiber grains with  $\langle 111 \rangle$  parallel to ND and the “other” grains which refers to any grain  
158 not belonging to the two previous fibers. A 15° tolerance was used to perform this  
159 classification.

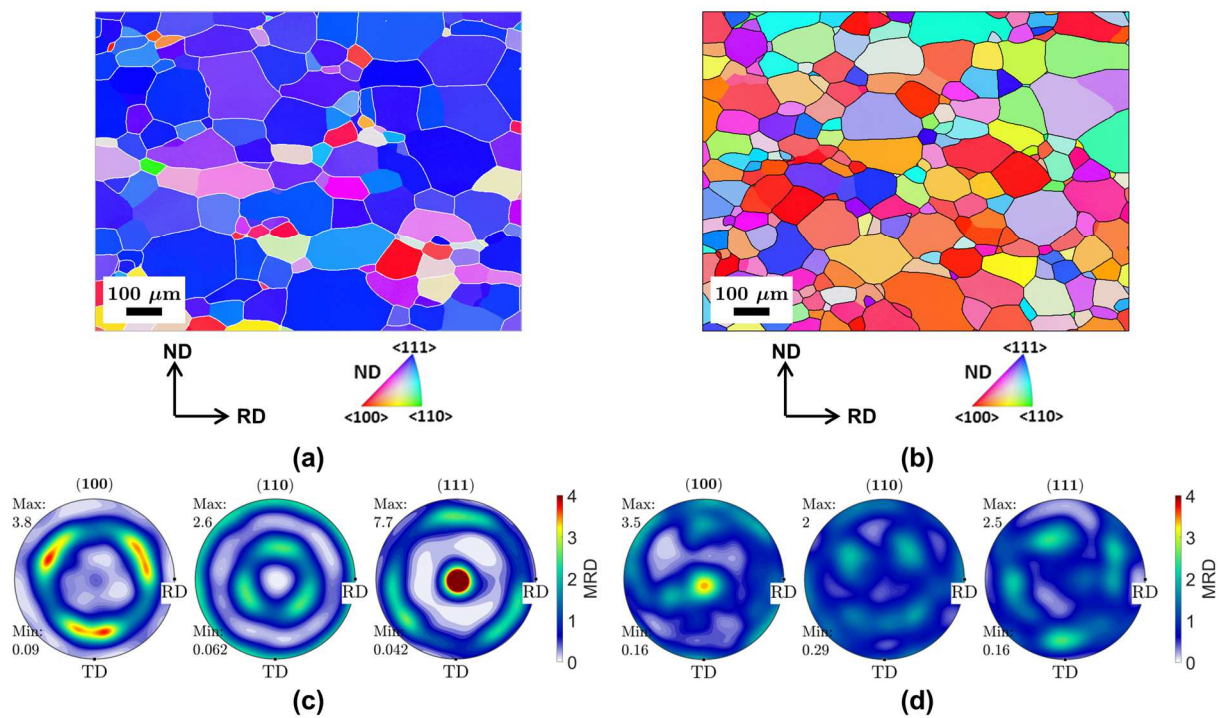
160 The  $\gamma$ -fiber is very intense at the center of the sheet (approximately 81% in grain surface  
161 fraction in Fig. 1a and see also the high intensity in {111} pole figure of Fig. 1c). For the  
162 microstructure in the quarter layer, the distribution of orientations is more homogeneous with  
163 a weaker  $\gamma$ -fiber and a stronger  $\theta$ -fiber as illustrated in Fig. 1b and 1d. This variation of

---

<sup>1</sup> The initial sheet was received in the form of a disc and the direction of the last rolling pass is unknown. The samples being taken from different locations on the sheet, the Radial Direction RD and the Transverse Direction TD are only indicative. On the other hand, ND stands for the real macroscopic normal direction of the initial sheet and is common to all the samples. Therefore, it is the reference direction used for all this study.

164 orientation can be attributed to the last stage of cold rolling before the recrystallization  
 165 treatment in the sheet forming process. Indeed, as observed both on bcc and fcc metals  
 166 [43,44], the effect of friction between the rolls and the sheet during cold rolling can induce  
 167 shear components near the sheet surface and then leads to through-thickness texture  
 168 heterogeneities.

169 The two observed microstructures show also a difference of grain size with larger grains in  
 170 the center than in the quarter layer, i.e. 156  $\mu\text{m}$  and 108  $\mu\text{m}$  for the average equivalent  
 171 diameter weighted by surface respectively (grains on the edges of EBSD maps were not  
 172 included in the calculation of grain sizes). Grains with an area smaller than 5 pixels were  
 173 excluded from analysis and the threshold disorientation angle of  $10^\circ$  was considered to  
 174 define grain boundaries.

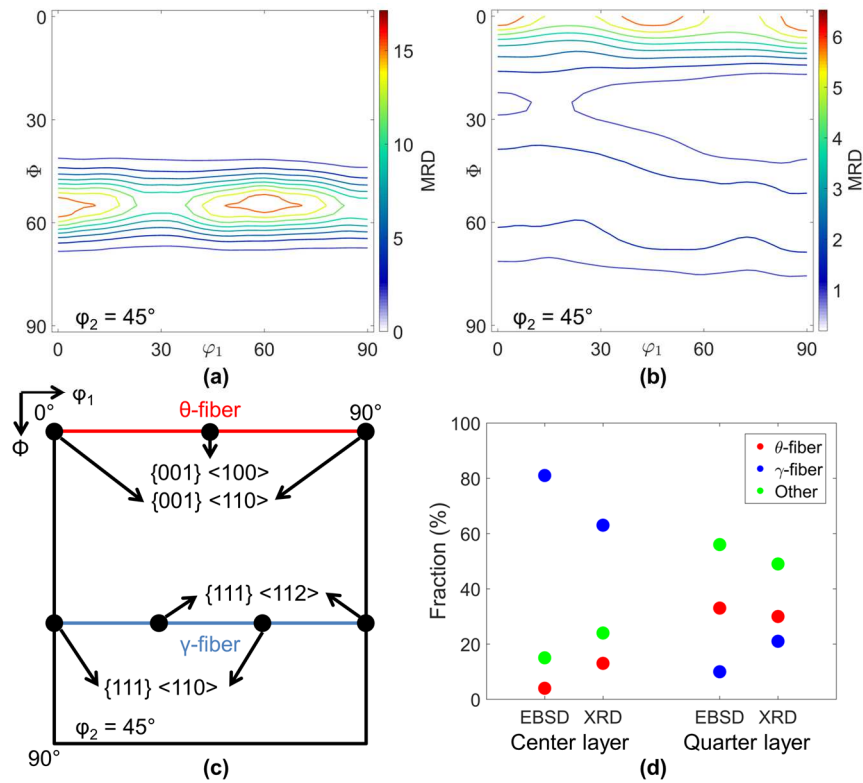


175  
 176 Fig. 1. Electron Back Scatter Diffraction (EBSD) orientation maps of the as-received material  
 177 for (a) the center of the sheet (samples used for compression) and (b) the quarter layer at  
 178 3 mm from the center (samples used for rolling) (Normal Direction ND projected onto the  
 179 standard triangle; RD: Radial Direction). Grain boundaries are plotted in white and black lines  
 180 for the center and the quarter layer of the sheet respectively ( $10^\circ$  disorientation angle  
 181 threshold). (c) and (d) corresponding pole figures for the two locations respectively (TD:  
 182 Transverse Direction).

183 In order to have a macrotexture information of the initial state, an X-ray diffraction instrument  
 184 (Panalytical<sup>®</sup> X'Pert<sup>3</sup>) with Cu K $\alpha$  radiation (45 kV/30 mA) was used at the center and at the

185 quarter layer of the sheet in the RD-TD (transverse direction) plane. Pole figures of {110},  
186 {200}, {211} and {222} were measured with a tilt angle from 0° to 69° with a step size of 3°.  
187 The orientation distribution functions (ODFs) were calculated from the above four poles  
188 figures using the MTEX toolbox [42] taking into account the noise effect and the ghost  
189 problem. As bcc materials often exhibit fiber textures after cold rolling or recrystallization, it is  
190 usual to represent the ODFs in the Euler space, in particular in the section  $\varphi_2 = 45^\circ$  where  $\theta$ -  
191 fiber and  $\gamma$ -fiber can be found as illustrated in Fig. 2c [1]. The macrotexture acquired at the  
192 center and at the quarter layer of the initial sheet are shown in Fig. 2a and 2b respectively.  
193 Results confirm the difference of texture between these two locations previously observed  
194 with the EBSD technique in Fig. 1c and 1d. {111} <110> orientations of  $\gamma$ -fiber appear to be  
195 very predominant at the center whereas {001} <100> and {001} <110> orientations of  $\theta$ -fiber  
196 prevail at the quarter layer but in a lesser extent with more uniformly distributed orientations.  
197 Moreover, the macrotexture obtained by XRD technique can be compared quantitatively to  
198 the microtexture obtained by EBSD technique as shown in Fig. 2d. For the quarter layer the  
199 two techniques display similar results concerning the fraction of each class of orientations.  
200 For the center more differences can be seen, in particular for the  $\gamma$ -fiber with a weaker  
201 fraction with the XRD technique ( $\approx 18\%$  less). This difference can result from the statistical  
202 relevance depending on the used technique or from spatial heterogeneities of texture in the  
203 initial sheet. As shown in Appendix A with EBSD maps ten times larger than the ones of  
204 Fig. 1, the dominance of the  $\gamma$ -fiber can vary by more than 10% depending on the position at  
205 the center of the sheet. This variation can explain the difference observed between XRD and  
206 EBSD results. Therefore, as these two techniques give close trends concerning the texture,  
207 only the EBSD technique is used in the following.





208  
 209 Fig. 2. ODF sections ( $\phi_2 = 45^\circ$ ) for (a) center and (b) quarter layers of the initial sheet; (c)  
 210 location of fibers and main texture components found in the section  $\phi_2 = 45^\circ$  of the Euler  
 211 space; (d) comparison of fraction of each class of orientations between EBSD (grain surface  
 212 fraction) and XRD techniques for center and quarter layers.

213

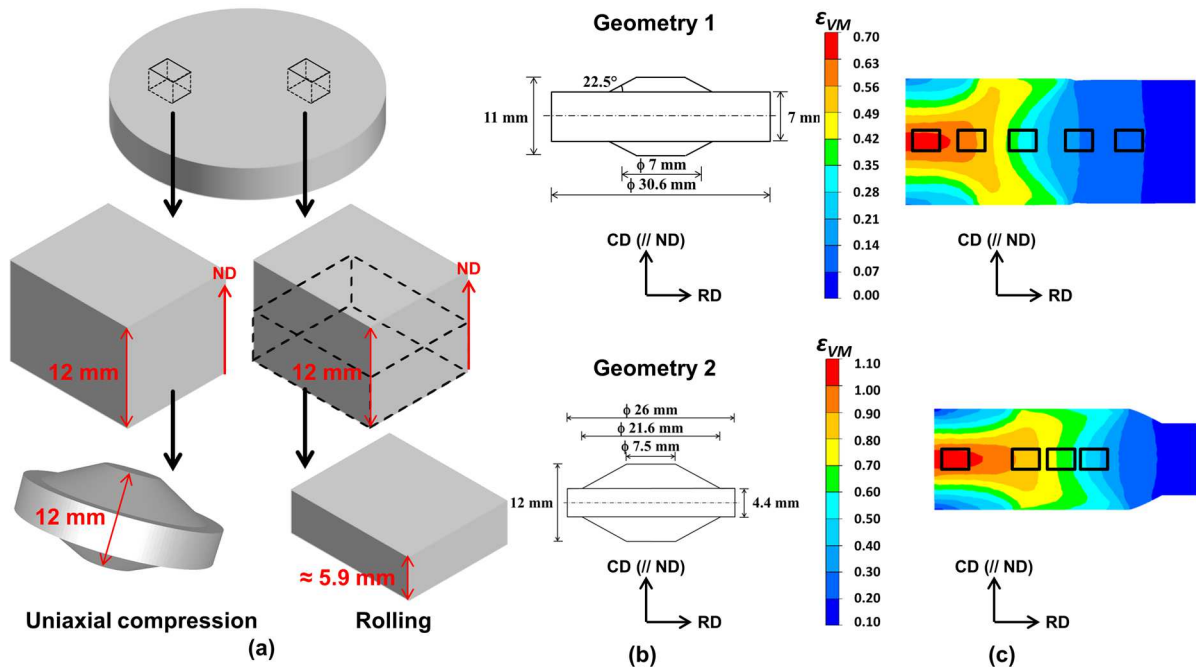
## 214 4. Deformed microstructure

### 215 4.1. Deformation conditions

216 Cold rolling and uniaxial compression tests were performed in order to study deformed  
 217 microstructures. For both deformation modes, parallelepipeds were taken from the initial  
 218 sheet at different locations as illustrated in Fig. 3a. For compression tests, samples were  
 219 machined in the total thickness of the parallelepipeds. Therefore, the initial state of the  
 220 compressed samples at the center is the one presented in Fig. 1a. The compression  
 221 direction CD is parallel to the normal direction ND of the initial sheet. In order to obtain a  
 222 strain gradient in one sample, two double-cone geometries, presented in Fig. 3b, were  
 223 chosen. The von Mises equivalent plastic strains ( $\epsilon_{VM}$ ) of the deformed samples were  
 224 determined using finite elements simulation with Forge<sup>®</sup> software as illustrated in Fig. 3c. The  
 225 constitutive laws used were taken from the work of Kerisit [45]. For the friction between the  
 226 tools and the sample, a Tresca law was used with a friction coefficient of 0.4, determined by  
 227 minimizing the difference of geometrical bulge between experimental and simulated

228 compression on cylinder. The two geometries used for this study lead to a  $\epsilon_{VM}$  that ranges  
 229 from 0.05 to 0.65 and from 0.15 to 1.09 respectively. The geometry 1 enables the  
 230 observation in detail of low strain case and higher strains can be reached with the  
 231 geometry 2.

232 For cold rolling, parallelepipeds were taken from the initial sheet and cut into two identical  
 233 halves (Fig. 3a). The obtained halves of around 5.9 mm thickness were used as samples for  
 234 rolling. Therefore, the initial state of the rolled samples at the center is the one presented in  
 235 Fig. 1b. The normal and the rolling directions of the rolling are parallel to the normal direction  
 236 ND and to the radial direction RD of the initial sheet respectively. With a 54 mm diameter  
 237 rolling mills and a rotation speed of 4 m/min five samples were cold rolled in several passes  
 238 (0.05 mm per pass) with lubricated rolls. Five different total thickness reductions of 38%,  
 239 45%, 58%, 68%, and 76% were obtained, resulting in five von Mises equivalent plastic  
 240 strains ( $\epsilon_{VM}$ ) respectively of 0.56, 0.70, 1.00, 1.33, 1.67 [46]. The ratio of the contact length  
 241 between the rolls and the sheet,  $l_c$ , to the sheet thickness,  $d$ , was kept between 1 and 5 to  
 242 ensure homogeneous deformation [44,47]. Moreover, a systematic rotation of the sample of  
 243 180° along the transverse direction was performed between the rolling passes.



244  
 245 Fig. 3. (a) Sketch representing where the samples for compression and rolling are taken from  
 246 the as-received sheet; (b) double-cone sample geometries; (c)  $\epsilon_{VM}$  in the CD-RD plane after  
 247 compression (CD: Compression Direction and RD: Radial Direction). Note that CD is the ND  
 248 of the initial sheet. Black rectangles show examined regions with SEM.

249 Deformed microstructures were characterized in terms of texture evolution and dislocation  
250 substructure development in the following sections. Observations are discussed in light of the  
251 difference of texture between the initial states of compressed and rolled samples and not in  
252 light of the difference of deformation mode. The influence of deformation mode is beyond the  
253 scope of this paper.

254

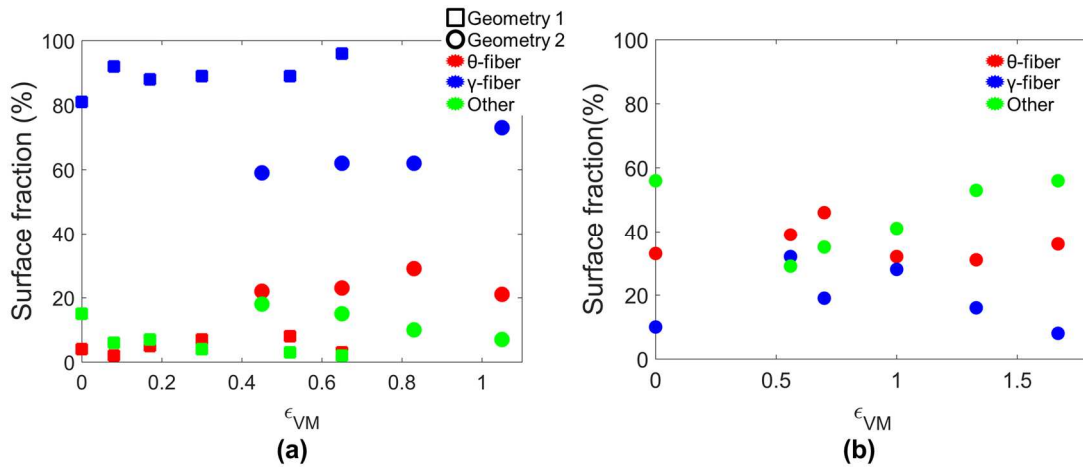
## 255 **4.2. Texture evolution**

256 EBSD maps of samples deformed by compression, with geometry 1 and 2 (Fig. 3b), and by  
257 rolling are presented in Appendices B, C and D respectively. The respective evolutions of  $\theta$ -  
258 fiber,  $\gamma$ -fiber and “other” orientations with strain are detailed quantitatively for the compressed  
259 and for the rolled samples in Fig. 4a and 4b respectively.

260 For both geometries of compression samples, the  $\gamma$ -fiber remains stable and very intense  
261 with strain. For geometry 1 samples, the surface fraction of  $\gamma$ -fiber grains is around 90% for  
262 all studied strains with a maximum of 96% for the highest strain. For geometry 2 samples,  
263 the surface fraction of  $\gamma$ -fiber grains is around 65% and of  $\theta$ -fiber grains around 20% for all  
264 strains. In both cases, plastic deformation does not seem to modify the crystallographic  
265 texture. The difference between the two cases is attributed to the initial state where the  
266 dominant  $\gamma$ -fiber is present between 70% and 80% depending on the location of EBSD maps  
267 as illustrated in section 3 and Appendix A. The observations made in Fig. 4a and the stability  
268 of the  $\gamma$ -fiber during uniaxial compression are consistent with the formation of fiber textures  
269 regularly found for bcc materials [48,49].

270 In the case of rolled samples, more variations of texture were observed. In the initial state the  
271  $\gamma$ -fiber and the  $\theta$ -fiber represent around 10% and 30% respectively as shown in Fig. 4b. For  
272  $\varepsilon_{VM} \approx 0.56$ , the  $\gamma$ -fiber is more intense and this can be surely related to the compression  
273 components at the center of the sheet. Above this strain, the intensity of  $\gamma$ -fiber continuously  
274 decreases with strain to the benefit of the two other classes of orientations, in particular the  
275 “Other” one which reaches more than 50% of the total surface. This decrease was also  
276 reported on previous experiment on pure Ta [50] and can be attributed to the same effect of  
277 friction between the rolls and the sheet already observed on starting material. Indeed, the  
278 superposition of added shear components to already present compression components  
279 might lead to a more randomized texture [43,44]. For small reductions, this effect is restricted  
280 to a layer near the surface and with increasing reduction it impacts deeper layers. For  
281 example, for  $\varepsilon_{VM} \approx 1.33$  and 1.67 the thickness of the samples is 1.9 mm and 1.4 mm  
282 respectively. Considering the width of EBSD maps (0.9 mm), a larger part of the

283 characterized surface contains zones impacted by the friction which tends to decrease the  
 284 intensity of  $\gamma$ -fiber and randomizes the texture. Furthermore, it can be noted that the  $\alpha$ -fiber,  
 285 another fiber generally composing the texture of cold rolled bcc materials [1,50], remains  
 286 quite stable during rolling in the present case with a surface fraction of  $\alpha$ -fiber grains between  
 287 32% and 46%.



288

289 Fig. 4. Evolution of  $\theta$ -fiber,  $\gamma$ -fiber and “other” orientations with strain for (a) compressed  
 290 samples and (b) rolled samples.

291

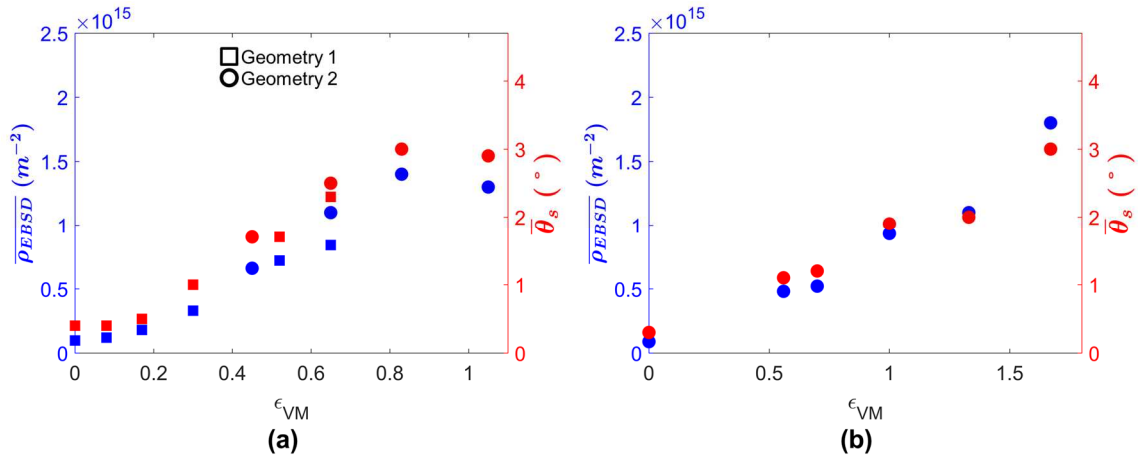
### 292 4.3. Deformation induced grain subdivision

#### 293 4.3.1. Intergranular analysis

294 Ashby’s classification distinguishes Statistically Stored Dislocations (SSDs) from  
 295 Geometrically Necessary Dislocations (GNDs) [51]. These two types have a distinct nature.  
 296 SSDs are created under homogeneous plastic deformation and do not imply orientation  
 297 gradient whereas GNDs are created for the accommodation of heterogeneous plastic  
 298 deformation and imply significant orientation gradient. As SSDs are redundant whereas  
 299 GNDs are not, the acquisition step size and the technique resolution for the orientation  
 300 measurement affects the distinction between them. Indeed, depending on the step size,  
 301 dislocation may be considered redundant or not. Hence, the use of a physically based  
 302 classification (SSDs and GNDs) to define quantities that are mainly distinguished by scale  
 303 effects and step size choice may be misleading. Therefore, dislocation densities estimated  
 304 from EBSD data are defined in the present paper as  $\rho_{EBSD}$ . In this paper, the method  
 305 proposed by Pantleon [52] and implemented by Seret et al. [53] was used. This method is  
 306 based on the continuum theory of dislocations with the determination of the Nye’s tensor  
 307 [54]. In order to avoid any misinterpretation that can be linked to the hypothesis of the used

308 method to estimate  $\rho_{EBSD}$ , the disorientation angle per segment  $\theta_s$  was used as a second  
 309 parameter to quantify intragranular disorientations induced by dislocation content [55]. This  
 310 parameter is the disorientation angle of each pair of adjacent pixels horizontally and vertically  
 311 on the overall EBSD map and the average value is then calculated.

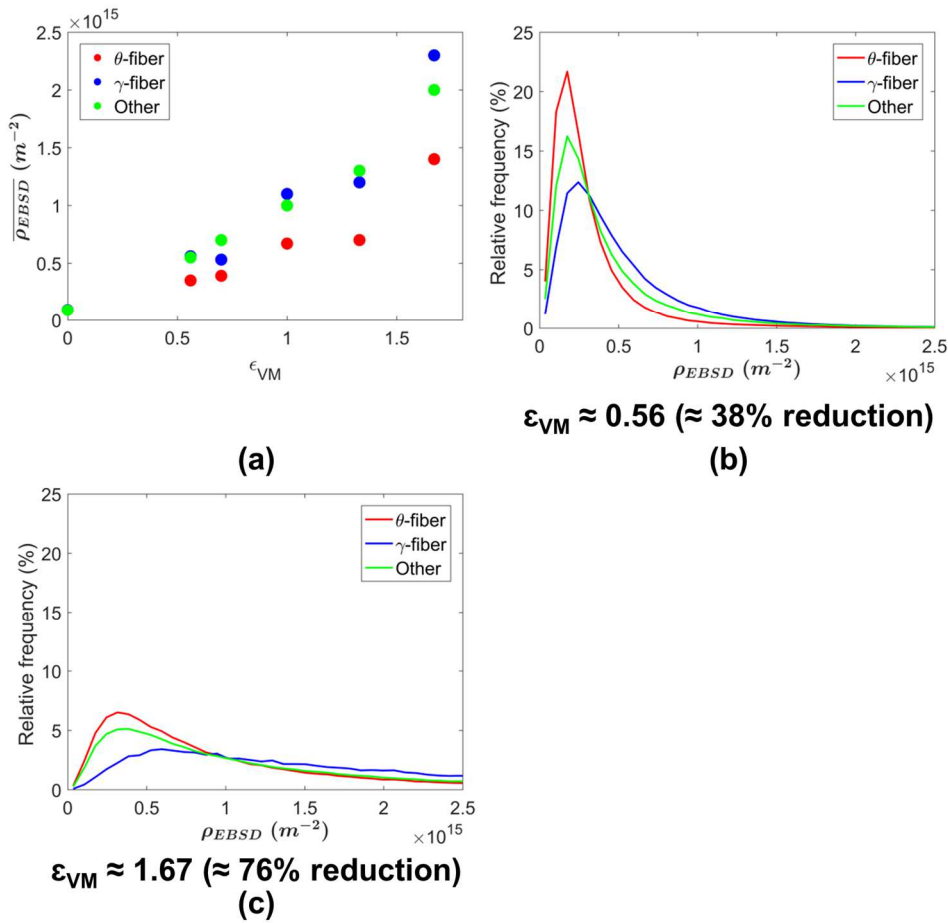
312 Using the EBSD maps presented in Appendices B, C and D,  $\rho_{EBSD}$  and  $\theta_s$  were calculated.  
 313 For the compressed samples, the evolutions of  $\overline{\rho_{EBSD}}$  and  $\overline{\theta_s}$  with strain are presented in  
 314 Fig. 5a. In the starting material,  $\overline{\rho_{EBSD}}$  and  $\overline{\theta_s}$  were determined as  $9.7 \times 10^{13} \text{ m}^{-2}$  and  $0.4^\circ$   
 315 respectively. As the material was deformed, the dislocation content quickly increases and  
 316 then seems to be saturating for further deformation with a plateau reaching  $1.3 \times 10^{15} \text{ m}^{-2}$  and  
 317  $2.9^\circ$  for  $\overline{\rho_{EBSD}}$  and  $\overline{\theta_s}$  respectively. Besides, the trend appears to be qualitatively similar for  
 318 both parameters.



319 Fig. 5. Evolution of  $\overline{\rho_{EBSD}}$  and  $\overline{\theta_s}$  with strain for (a) compressed samples and (b) rolled  
 320 samples.  
 321

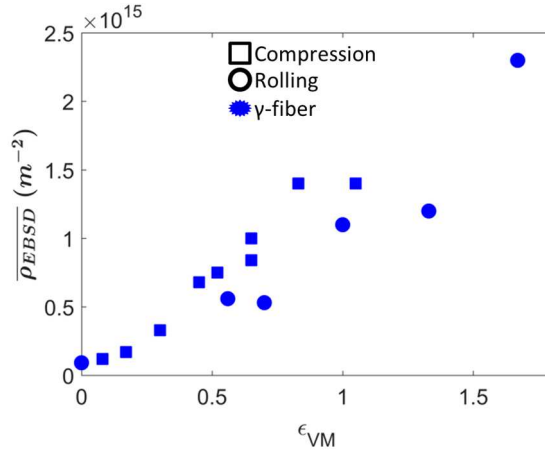
322  
 323 For the rolled samples, the evolutions of  $\overline{\rho_{EBSD}}$  and  $\overline{\theta_s}$  with strain are presented in Fig. 5b.  
 324 Similarly to the compression case these values increase with deformation as expected, but  
 325 no apparent saturation is observed for this case. It is worth noticing that the increase in the  
 326 case of rolling (Fig. 5b) is slower than in the case of compression (Fig. 5a). This difference is  
 327 probably induced by the differences in the crystallographic orientations. Indeed, for the case  
 328 of compression, the material at the initial state has between 70% and 80% of  $\gamma$ -fiber grains.  
 329 On the contrary, for the case of rolling the material at the initial state has around 10% and  
 330 30% of  $\gamma$ -fiber and  $\theta$ -fiber grains respectively. Therefore, the evolutions of  $\overline{\rho_{EBSD}}$  and  $\overline{\theta_s}$   
 331 with strain depend on the crystallographic orientation. To examine this influence, all the pixels of  
 332 grains belonging to each class of orientations are gathered and then  $\overline{\rho_{EBSD}}$  and  $\overline{\theta_s}$  are  
 333 calculated for each class independently. As  $\overline{\rho_{EBSD}}$  and  $\overline{\theta_s}$  gave similar results for the  
 334 compression and the rolling cases, the study of the orientation effect is depicted only with  
 335  $\overline{\rho_{EBSD}}$  in the following. The evolutions of  $\overline{\rho_{EBSD}}$  with strain for  $\theta$ -fiber,  $\gamma$ -fiber and “other”-

336 oriented grains are shown in Fig. 6a and the distributions of  $\rho_{EBSD}$  for each class of  
 337 orientations are presented in Fig. 6b and 6c for  $\epsilon_{VM} \approx 0.56$  and 1.67 respectively. The  
 338 evolution of the dislocation content differs between each class of orientations.  $\overline{\rho_{EBSD}}$  is higher  
 339 for the  $\gamma$ -fiber grains than for the  $\theta$ -fiber grains of approximately 60% and the increase seems  
 340 also faster for the  $\gamma$ -fiber grains. Moreover, the distributions appear also to be wider for the  $\gamma$ -  
 341 fiber grains with a shift to the right with increasing strain, illustrating a distinct intragranular  
 342 behavior between these two fibers. These results are consistent with the numerous  
 343 investigations on the influence of crystallographic orientation on deformation performed on  
 344 bcc materials such as Fe [33,35], steel [34,56] or Ta [37–39,57] for instance.



345  
 346 Fig. 6. (a) Evolution of  $\overline{\rho_{EBSD}}$  with strain for each class of orientations; distribution of  $\rho_{EBSD}$  for  
 347 each class of orientations for  $\epsilon_{VM} \approx$  (b) 0.56 and (c) 1.67.

348  
 349 As it can be observed in Fig. 5a and Fig. 5b, the evolutions of  $\overline{\rho_{EBSD}}$  are different between the  
 350 compression case and the rolling case. However, when only the  $\gamma$ -fiber grains are  
 351 considered, the difference between these two evolutions is lower at the studied scale as  
 352 presented in Fig. 7. Despite this observation at the macroscopic scale, the behavior of the  $\gamma$ -  
 353 fiber grains at the dislocation substructure scale is very different between these two cases as  
 354 illustrated later in the following section.

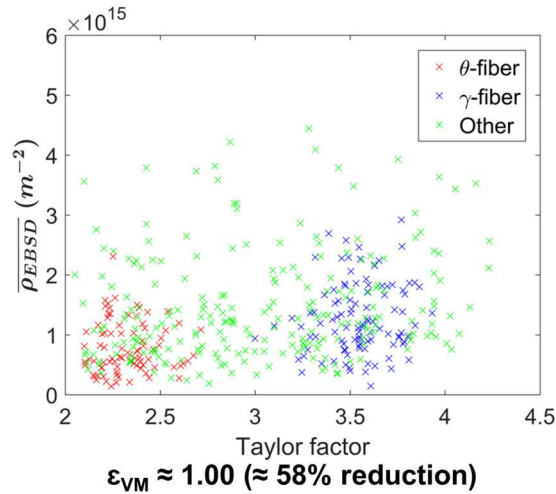


355

356 Fig. 7. Evolution of  $\overline{\rho_{EBSD}}$  with strain for compressed samples (the two geometries are taken  
 357 together) and rolled samples for the  $\gamma$ -fiber grains.

358

359 The Taylor factor describes the slip systems activity during plastic deformation and is related  
 360 to the amount of work required to deform grains for a given strain. As it is quite difficult to  
 361 determine which specific slip system is activated for each grain, the typical slip systems for  
 362 bcc materials [1] are employed for the calculation of the Taylor factor ( $\{110\} \langle 111 \rangle$ ,  
 363  $\{112\} \langle 111 \rangle$ ,  $\{123\} \langle 111 \rangle$ ) and it is assumed that the critical resolved shear stress is the  
 364 same for all slip systems. The calculation is performed with the MTEX toolbox [42]. The  
 365 Taylor factor for the  $\gamma$ -fiber, the  $\theta$ -fiber and “other”-oriented grains are 3.5, 2.4 and 3  
 366 respectively. Those values are in agreement with the previous disparities of  $\overline{\rho_{EBSD}}$  between  
 367 the three sets of grains (Fig. 6a) highlighting the difference of behavior during plastic  
 368 deformation. However, when each grain is considered separately no direct correlation  
 369 between the Taylor factor and  $\overline{\rho_{EBSD}}$  was observed for none of the deformed states as  
 370 reported for  $\epsilon_{VM} \approx 1.00$  in Fig. 8. Weak and partial correlations between the Taylor factor and  
 371 the dislocation content have previously been reported [58–60]. On the basis of those  
 372 observations, the Taylor factor can explain global trends between each class of orientations  
 373 but is not relevant to predict the intragranular deformation of grains. Indeed, the local  
 374 boundary conditions on each grain are different from the macroscopic ones because of the  
 375 different neighboring grains’ behavior.



376

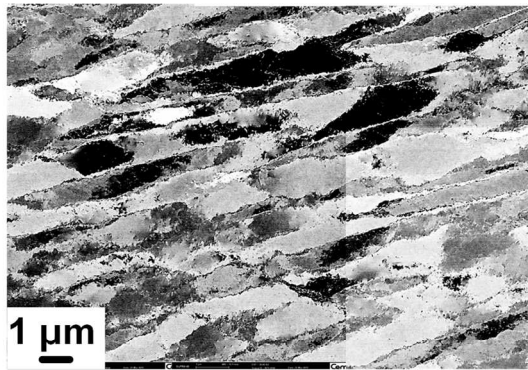
377 Fig. 8.  $\overline{\rho_{EBSD}}$  per grain vs Taylor factor plot for rolled sample for  $\epsilon_{VM} \approx 1.00$ .

378

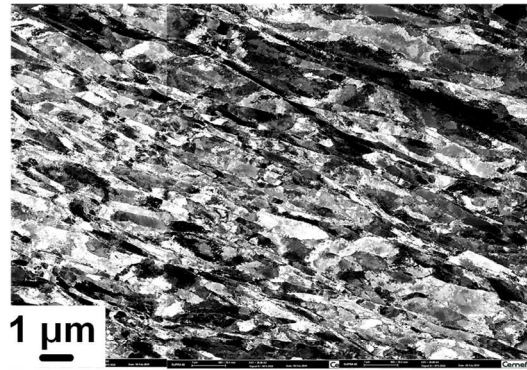
### 379 4.3.2. Dislocation substructures analysis

380 The deformed microstructures were observed at a lower scale to analyze the dislocation  
 381 substructures. A first direct qualitative observation was realized with the ECCI technique with  
 382 a specific investigation on the  $\theta$ -fiber and the  $\gamma$ -fiber grains. ECCI micrographs performed in  
 383 the interior of the grains in compressed and rolled microstructures are shown in Fig. 9 and 10  
 384 respectively.

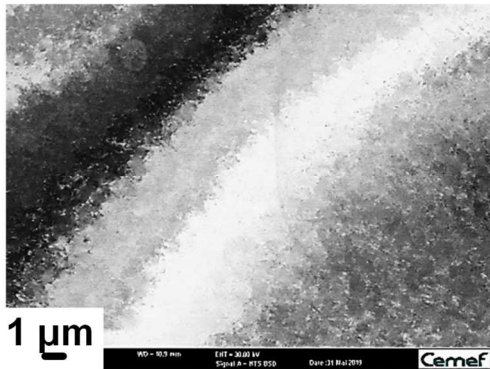




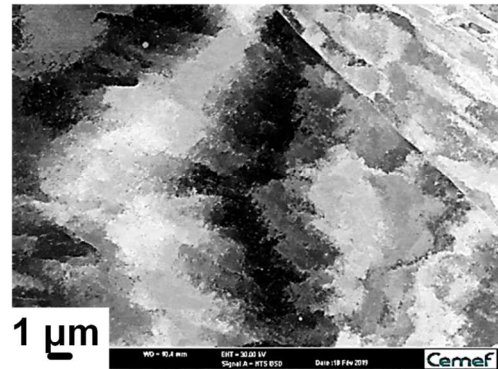
$\epsilon_{VM} \approx 0.30 \pm 0.07$   
(a)



$\epsilon_{VM} \approx 1.05 \pm 0.02$   
(b)

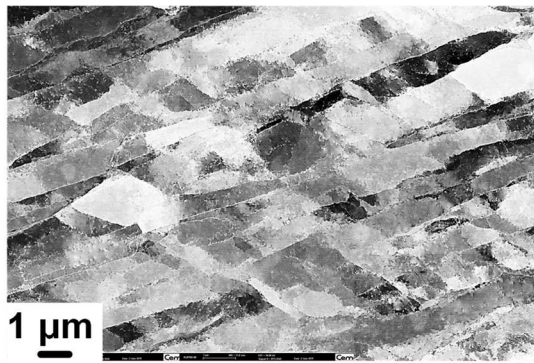


$\epsilon_{VM} \approx 0.30 \pm 0.07$   
(c)

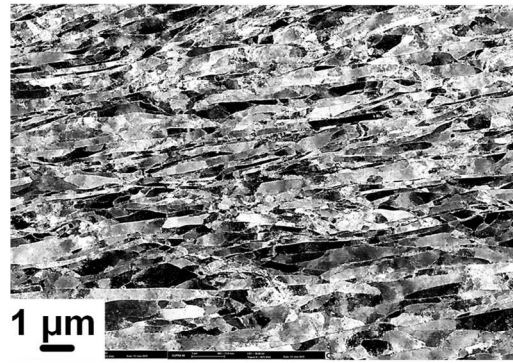


$\epsilon_{VM} \approx 1.05 \pm 0.02$   
(d)

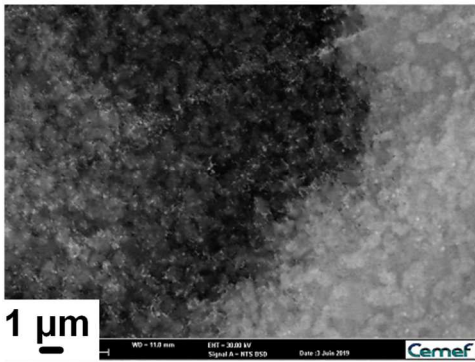
385  
386 Fig. 9. ECCL micrographs on (a) & (b)  $\gamma$ -fiber grains and (c) & (d)  $\theta$ -fiber grains for  $\epsilon_{VM} \approx 0.30$   
387 and 1.05 respectively for compressed samples (the compression direction is vertical).



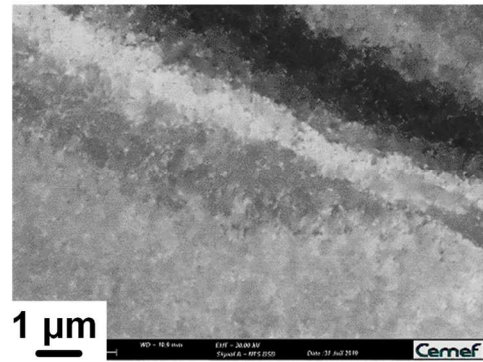
$\epsilon_{VM} \approx 0.56$  ( $\approx 38\%$  reduction)  
(a)



$\epsilon_{VM} \approx 1.67$  ( $\approx 76\%$  reduction)  
(b)



$\epsilon_{VM} \approx 0.56$  ( $\approx 38\%$  reduction)  
(c)



$\epsilon_{VM} \approx 1.67$  ( $\approx 76\%$  reduction)  
(d)

388

389 Fig. 10. ECCI micrographs on (a) & (b)  $\gamma$ -fiber grains and (c) & (d)  $\theta$ -fiber grains for  
390  $\epsilon_{VM} \approx 0.56$  and 1.67 respectively for rolled samples (the rolling normal direction is vertical).

391 For the two cases, there is a distinct substructure development between the two fibers.  
392 For the  $\gamma$ -fiber grains (Fig. 9a and b and Fig. 10a and b) dislocation substructures are well  
393 defined even for the smallest deformation  $\epsilon_{VM} \approx 0.30$ . For small strains (Fig. 9a and 10a)  
394 evidence of dislocations entanglement can be observed in the subboundaries and there are  
395 some traces of dislocations inside subgrains. For high strains (Fig. 9b and 10b) thinner  
396 subboundaries are better defined and less traces of dislocations inside subgrains are  
397 observed. In addition, the subgrain size is observed to decrease with increasing strain which  
398 is in coherence with previous results as reported in section 1. For the rolled samples  
399 (Fig. 10a and b) the subboundaries look also gradually more aligned parallel to the rolling  
400 direction with increasing strain which is consistent with previous investigations on Al and Ni  
401 [22,28,36].

402 For the  $\theta$ -fiber grains (Fig. 9c and d and Fig. 10c and d) no dislocation substructures can be  
403 observed even for the highest strain  $\epsilon_{VM} \approx 1.67$  with just few developed subboundaries. This  
404 observation is in agreement with the orientation dependence of intragranular heterogeneities

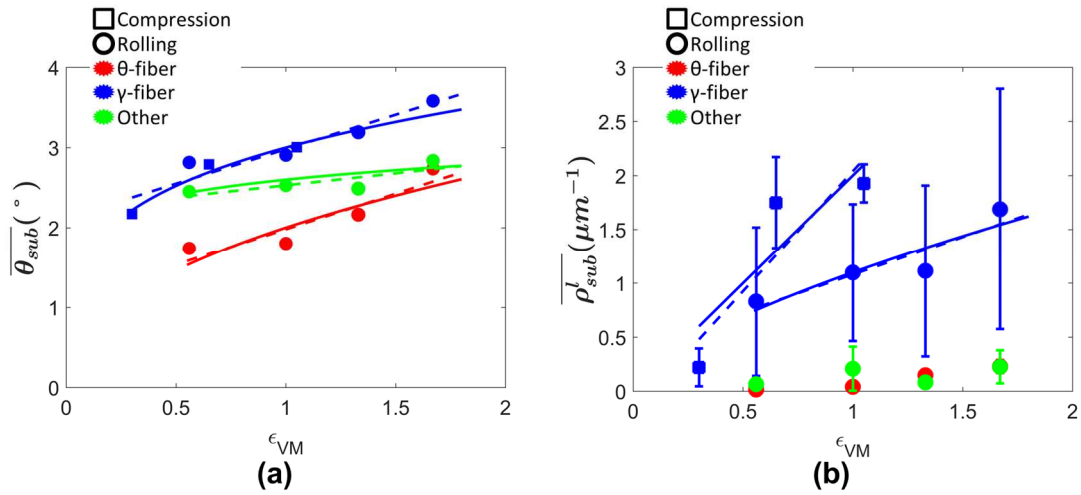
405 noticed previously at larger scale (section 4.3.1). It can be concluded that even if  $\overline{\rho_{EBSD}}$  of  $\theta$ -  
406 fiber grains increases with strain (Fig. 6a), this increase does not involve sharper  
407 substructure development. Therefore, the evolution of the dislocation content and their  
408 spatial distribution within substructures are different between the  $\theta$ -fiber and the  $\gamma$ -fiber  
409 grains.

410 EBSD maps with an acquisition step size of 90 nm were performed in the interior of the  
411 grains to investigate quantitatively the dislocation substructures. Acquisitions were done the  
412 most possible at the center of grains to have results more representative to the  
413 crystallographic orientation and less affected by the proximity to grain boundaries. In order to  
414 have statistical relevance, EBSD maps were acquired on several grains for each strain level  
415 and for each class of orientations. Only the  $\gamma$ -fiber grains were investigated for the  
416 compression case because of the very high intensity of this fiber. The number of examined  
417 grains and the total surface observed with EBSD for each case are summarized in Table 1.  
418 The segments with a disorientation angle between  $1.5^\circ$  and  $10^\circ$  are considered to be induced  
419 by dislocation subboundaries and so are denoted as  $\theta_{sub}$ . In order to describe the  
420 substructure development, two parameters were employed: the average disorientation angle  
421 of subboundaries  $\overline{\theta_{sub}}$  ( $^\circ$ ) and the average linear density of subboundaries  $\overline{\rho_{sub}^l}$  ( $\mu\text{m}^{-1}$ ). The  
422 latter is calculated with the division of the sum of subboundary length by the total observed  
423 surface.

424 Table 1: Number of examined grains and total surface observed with EBSD for the  
 425 observation at dislocation substructure scale.

Deformation mode	Strain level ( $\epsilon_{VM}$ )	Class of orientations	Number of examined grains	Total surface observed with EBSD ( $\mu\text{m}^2$ )	Average estimated orientation gradient ( $^\circ/\mu\text{m}$ )
Compression	0.30	$\gamma$ -fiber	6	21891	0.07
	0.65	$\gamma$ -fiber	6	17602	0.12
	1.05	$\gamma$ -fiber	8	24177	0.17
Rolling	0.56	$\theta$ -fiber	2	2990	0.23
	1.00	$\theta$ -fiber	2	2256	0.28
	1.33	$\theta$ -fiber	4	6957	0.45
	1.67	$\theta$ -fiber	4	2080	0.91
	0.56	$\gamma$ -fiber	5	15037	0.22
	1.00	$\gamma$ -fiber	6	9278	0.23
	1.33	$\gamma$ -fiber	6	12305	0.32
	1.67	$\gamma$ -fiber	7	3631	0.93
	0.56	"Other" class	5	10104	0.27
	1.00	"Other" class	4	4454	0.39
	1.33	"Other" class	4	5265	0.52
	1.67	"Other" class	5	3091	0.73

426



427

428 Fig. 11. Evolution of (a)  $\overline{\theta_{sub}}$  and (b)  $\overline{\rho_{sub}^l}$  with strain for each class of orientations.

429 Represented standard deviations are weighted by the observed surface of each EBSD map.

430 The dashed and full lines are least squares fits with linear and power laws of Eq. (1), (2) and

431 (3) for  $\overline{\theta_{sub}}$  and Eq. (4) and (5) for  $\overline{\rho_{sub}^l}$ .

432 The evolution of  $\overline{\theta}_{sub}$  with strain for each class of orientations is illustrated in Fig. 11a. The  
 433 orientation dependence of dislocation substructure development observed previously with  
 434 ECCI technique is encountered again. Indeed, the subboundaries are more misoriented with  
 435 increasing strain and the disorientation is always higher in the  $\gamma$ -fiber grains than in the  
 436 grains of the two other classes. No saturation of the subboundaries' disorientation is  
 437 observed in the present conditions. Deformation induced subboundaries can be classified  
 438 into Incidental Dislocation Boundaries (IDBs) and Geometrically Necessary Boundaries  
 439 (GNBs) [23]. For both of these boundaries, the disorientation angle increases with strain but  
 440 in a distinct way. Pantleon proposed semi-empirical laws to describe the evolution of the  
 441 disorientation angle for the two types of boundaries [61,62]. He applied these two laws on  
 442 data obtained on cold rolled pure Al or Al alloy [12,63]. However, these laws might not be  
 443 suitable to bcc materials because the fit with the present experimental data does not show  
 444 satisfactory results. As a lower disorientation threshold is considered in the present case, the  
 445 majority of IDBs are ignored. Consequently, the characterized subboundaries are probably  
 446 GNBs. In the present case, least squares fits with a linear and a power law give suitable  
 447 results:

448 -  $\theta$ -fiber grains:  $\overline{\theta}_{sub} (\text{°}) = 0.89 \varepsilon_{VM} + 1.09$  or  $\overline{\theta}_{sub} (\text{°}) = 2.02 \varepsilon_{VM}^{0.45}$  (1)

449 -  $\gamma$ -fiber grains:  $\overline{\theta}_{sub} (\text{°}) = 0.86 \varepsilon_{VM} + 2.12$  or  $\overline{\theta}_{sub} (\text{°}) = 3.04 \varepsilon_{VM}^{0.25}$  (2)

450 - "Other"-oriented grains:  $\overline{\theta}_{sub} (\text{°}) = 0.30 \varepsilon_{VM} + 2.23$  or  $\overline{\theta}_{sub} (\text{°}) = 2.56 \varepsilon_{VM}^{0.11}$  (3)

451 The differences between these equations show clearly that there is no universal law to  
 452 describe the evolution of the subboundaries disorientation for whatever crystallographic  
 453 orientation. The power law coefficient is close to 0.5 for the  $\theta$ -fiber grains (Eq. (1)) which  
 454 corresponds to the law proposed by Pantleon [61,62] for IDBs. This result allows to speculate  
 455 that dislocation subboundaries close to IDBs can appear in the  $\theta$ -fiber grains, as already  
 456 supposed in previous work [38]. Experimental results for higher strains are necessary in  
 457 order to decide which type of law fits better: a linear or a power law.

458 On the other hand, more substructures are developed in the  $\gamma$ -fiber grains as shown in  
 459 Fig. 11b. The quantity of subboundaries remains very low even for high strains for the grains  
 460 of the two other classes of orientations. Focusing on the  $\gamma$ -fiber grains,  $\overline{\rho}_{sub}^l$  shows distinct  
 461 evolution between the compression case and the rolling case. For rolled samples,  $\overline{\rho}_{sub}^l$   
 462 increases continuously with strain. For compressed samples, more subboundaries are  
 463 developed in the  $\gamma$ -fiber grains and  $\overline{\rho}_{sub}^l$  appears to quickly saturate around  $2 \mu\text{m}^{-1}$  for  
 464  $\varepsilon_{VM} \approx 0.65$ . Higher strains obtained by compression would confirm this saturation. The  
 465 present results for the compressed  $\gamma$ -fiber grains are consistent with those previously

466 reported for cold rolled pure nickel [64]. The evolutions of the grain size for both cases are  
 467 similar. This observation indicates that the different evolution of  $\overline{\rho_{sub}^l}$  in the  $\gamma$ -fiber grains for  
 468 compression and rolling is not because of subboundaries transforming into boundaries with  
 469 disorientation angles higher than the upper threshold of  $10^\circ$ .

470 Besides, the development of dislocation substructures seems more homogeneous during the  
 471 compression deformation than during the rolling deformation since the distribution of  $\rho_{sub}^l$   
 472 values spreads on larger range for the latter. Those two observations are probably related to  
 473 the strong  $\gamma$ -fiber texture observed in the samples used for compression (Fig. 4a). Indeed,  
 474 the  $\gamma$ -fiber grains represent between 70% and 80% of the initial state for the compressed  
 475 samples and no more than 10% of the one for the rolled samples. Therefore, because of the  
 476 crystal plasticity and the interactions between the different types of grains, the compressed  
 477 samples present more homogeneous dislocation subboundaries with less spread as  
 478 observed in Fig. 11b. This homogeneity is the probable reason behind the fast increase and  
 479 the saturation of  $\overline{\rho_{sub}^l}$  for the case of compressed samples. In the case of rolled samples,  
 480 dislocations subboundaries continue to form even at high strains because of the  
 481 heterogeneity of the subboundaries. Hutchinson reported on cold rolled steel [56] a similar  
 482 impact of the neighboring environment on the grain deformation. The case of a “hard” grain  
 483 with a high Taylor factor adjacent to a “soft” grain with a low Taylor factor was investigated.  
 484 The hard grain, which has a higher flow stress, tends to resist deformation whereas the soft  
 485 grain has to bend around this grain. This leads also to a weakening of the texture as the  
 486 orientation of the soft grain will be spread. This case can be transposed to the present rolling  
 487 case:  $\gamma$ -fiber grains are quite often surrounded by softer  $\theta$ -fiber and “other”-oriented grains  
 488 which will deform more easily as can be seen with EBSD maps of Appendix D.

489 As for  $\overline{\theta_{sub}}$  previously, the evolution of  $\overline{\rho_{sub}^l}$  with strain can be modelled. Considering the low  
 490 values for  $\theta$ -fiber and “other” grains, the modelling was only done for  $\gamma$ -fiber grains.  
 491 Moreover, as the evolutions are different between compression and rolling cases, distinct  
 492 laws were determined for each case. Least squares fits with a linear and a power law give  
 493 suitable results:

494 - Compressed  $\gamma$ -fiber grains:  $\overline{\rho_{sub}^l} (\mu m^{-1}) = 2,23 \varepsilon_{VM} - 0,19$  or  $\overline{\rho_{sub}^l} (\mu m^{-1}) = 2,00 \varepsilon_{VM}^{1,01}$  (4)

495 - Rolled  $\gamma$ -fiber grains:  $\overline{\rho_{sub}^l} (\mu m^{-1}) = 0,70 \varepsilon_{VM} + 0,38$  or  $\overline{\rho_{sub}^l} (\mu m^{-1}) = 1,10 \varepsilon_{VM}^{0,66}$  (5)

496 Values of  $\overline{\rho_{sub}^l}$  can be related to the substructure sizes with stereographic considerations and  
 497 with the hypothesis that the substructures are spheres, have all the same size and are  
 498 uniformly distributed along the surface [65,66]. Thus, the average sizes can be determined  
 499 with the following equation:

$$\overline{d_{sub}} = \frac{2}{3} \pi \frac{1}{\overline{\rho_{sub}^l}} \quad (6)$$

501 where  $\overline{d_{sub}}$  is the average substructure equivalent diameter ( $\mu\text{m}$ ) and  $\overline{\rho_{sub}^l}$  is the average  
 502 linear density of subboundaries ( $\mu\text{m}^{-1}$ ).

503 This calculation was performed only for  $\gamma$ -fiber grains because values of  $\overline{\rho_{sub}^l}$  were very low  
 504 (the hypothesis of uniform distribution is not valid) for the other grains and led to non-physical  
 505 substructure sizes of several tens of  $\mu\text{m}$ . For the same reason, the first strain for the  
 506 compression case ( $\varepsilon_{VM} \approx 0.30$ ) was not considered so only the rolling case was modelled.  
 507 Least squares fits of experimental points with a linear and a power law give correct results:

$$508 \quad - \quad \text{Rolled } \gamma\text{-fiber grains: } \overline{d_{sub}} (\mu\text{m}) = -1.06 \varepsilon_{VM} + 3.10 \text{ or } \overline{d_{sub}} (\mu\text{m}) = 1.90 \varepsilon_{VM}^{-0.51} \quad (7)$$

509

510

### 4.3.3. Long-range orientation gradients analysis

511 Although no subboundaries (abrupt disorientations) are clearly formed in the  $\theta$ -fiber grains in  
 512 the ECCI micrographs (Fig. 9c and d and Fig. 10c and d), orientation contrasts are observed  
 513 in these grains with progressive transitions from white to black regions and might imply the  
 514 presence of long-range orientation gradients. The softer  $\theta$ -fiber grains are prone to be  
 515 subject to internal bending due to the neighboring grains with different deformation behaviors  
 516 inducing long-range orientation gradients. As observed on cold rolled pure tantalum [67], the  
 517  $\theta$ -fiber grains can even split due to the interaction with neighboring  $\gamma$ -fiber grains. In contrast,  
 518 in the  $\gamma$ -fiber grains, the disorientations across subboundaries tend to alternate with no  
 519 significant long-range orientation gradient as observed on cold rolled pure aluminum [36].

520 For the analysis of orientation gradients, EBSD maps of Table 1 were used. Disorientation  
 521 profiles passing through the center of the map at every  $3^\circ$  were plotted for each EBSD map  
 522 (i.e. 60 profiles per map). The evolution of the disorientation along each profile was least  
 523 squares fitted with a linear law and the slope was considered as the orientation gradient.  
 524 Since there is no guarantee that one of the profiles is in the same direction as the highest  
 525 orientation gradient, the maximal value was considered as the closest one to the real  
 526 orientation gradient (this value underestimates the real orientation gradient). For each case  
 527 of Table 1, the average value (weighted by the surface of EBSD map) of the estimated  
 528 orientation gradient is calculated (see the right column of Table 1).

529 For each class of orientations, the average orientation gradient increases with strain as  
 530 expected. Long-range orientation gradients tend to develop continuously with strain in  $\theta$ -fiber  
 531 grains whereas in  $\gamma$ -fiber grains gradients become more significant for high strain  
 532 ( $\varepsilon_{VM} \approx 1.67$ ), which might reveal a beginning of grain fragmentation. Therefore, although

533 orientation gradients are more apparent in  $\theta$ -fiber grains with the ECCI micrographs (Fig. 9c  
534 and d and Fig. 10c and d), they are not higher than the ones in  $\gamma$ -fiber grains. Moreover,  
535 orientation gradients in  $\gamma$ -fiber grains appear to be higher for the rolling case than for the  
536 compression case. This difference is most probably due to the more varied neighborhood of  
537  $\gamma$ -fiber grains in rolled samples.

538

## 539 **5. Conclusion**

540 In this study, a quantitative analysis of cold deformed pure tantalum microstructure is  
541 presented. Two deformation modes, compression and rolling, each applied on a different  
542 initial state were employed to access the influence of crystallographic orientation. The  
543 orientation dependence of deformation was emphasized with two different scales of  
544 observation with SEM. The following conclusions can be drawn from the present analysis:

- 545 • During compression, the  $\gamma$ -fiber seems to be stable. For the case of rolling, the friction  
546 causing shear components leads to a decrease in the  $\gamma$ -fiber fraction.
- 547 • At grain scale, intragranular heterogeneities were evaluated with the dislocation  
548 density  $\rho_{EBSD}$ .  $\gamma$ -fiber grains have always higher values than  $\theta$ -fiber and “other”-  
549 oriented grains. At this scale, there is no significant difference in  $\rho_{EBSD}$  values  
550 between compressed and rolled  $\gamma$ -fiber grains.
- 551 • At dislocation substructure scale, this orientation dependence was also pointed out  
552 with developed substructures in the interior of  $\gamma$ -fiber grains and almost no  
553 substructures in the  $\theta$ -fiber grains. Quantitative characterization of substructure  
554 development was performed and models describing the evolution of the average  
555 disorientation and size of substructures were proposed for the  $\gamma$ -fiber grains. At this  
556 scale, the difference of substructure development between compressed and rolled  $\gamma$ -  
557 fiber grains is significant.
- 558 • When the  $\gamma$ -fiber is weakly present (no more than 30% for the rolling case), the  
559 substructure development is quite heterogeneous and the substructure disorientation  
560 and size increase continuously with increasing strain. However, when the  $\gamma$ -fiber is  
561 dominant (more than 65% for the compression case), a very fast saturation of  
562 subboundaries density was observed. In such conditions, subboundaries are formed  
563 for small strains and only their disorientation increases for further strains.
- 564 • Intragranular orientation gradients were analyzed. For the rolling case, long-range  
565 orientation gradients tend to develop continuously with strain in  $\theta$ -fiber grains  
566 whereas in  $\gamma$ -fiber grains gradients become substantial for high strain ( $\epsilon_{VM} \approx 1.67$ ).



567 For both cases the orientation gradients were very close. Orientation gradients in  $\gamma$ -  
568 fiber grains are higher in rolled samples than in compressed samples as the  
569 neighborhood of  $\gamma$ -fiber grains is more varied for the former case.

570 One of the main conclusions of the present work is that describing the deformed  
571 microstructure through dislocation density is not enough. Therefore, in order to study and  
572 model thermally activated physical mechanisms driven by stored energy induced by  
573 dislocations, like recovery and recrystallization, it is necessary to quantify dislocation  
574 substructures.

575

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578 their help in performing sample preparation, deformation experiments, microscopy analyzes  
579 and XRD characterization respectively. The authors are also very grateful to Rémy Besnard  
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581 and to CEA DAM Valduc for funding.

582

## 583 **Data availability**

584 The raw/processed data required to reproduce these findings cannot be shared at this time  
585 as the data also forms part of an ongoing study.

586

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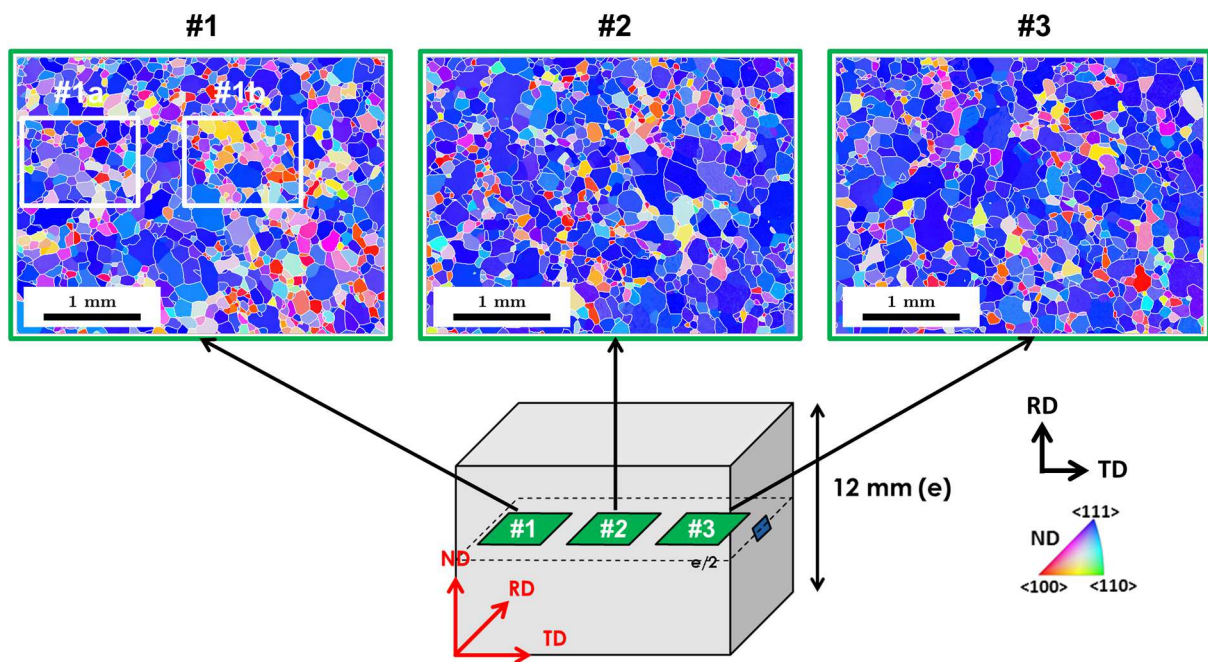
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769 **Appendices**

770 **Appendix A: Variations of texture at the center of the initial sheet**

771 The different distributions of  $\gamma$ -fiber between EBSD and XRD techniques (Fig. 2d) and also  
772 between the two geometries of compression samples (Fig. 4) might stem from spatial  
773 heterogeneities. In order to verify this possibility, the microstructure at the center of the initial  
774 sheet was characterized in the plane radial direction RD/transverse direction TD as  
775 illustrated in Fig. A.1. Three EBSD maps ten times larger than the ones used in this study  
776 (Fig. 1a for example) were performed at three different random locations with an acquisition  
777 step size of  $3.45\ \mu\text{m}$  over a rectangular area of  $3.80\ \text{mm} \times 2.85\ \text{mm}$ .

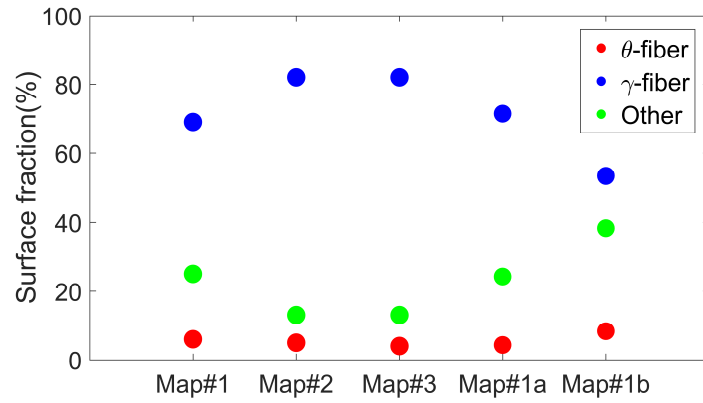


778

779 Fig. A.1. EBSD orientation maps acquired at the center of the initial sheet (ND projected onto  
780 the standard triangle). Grain boundaries are plotted in white lines ( $10^\circ$  disorientation angle  
781 threshold). Schematic blue rectangle on the right side of the schema and white rectangles on  
782 EBSD map #1 represent the EBSD map size used in this study as the one of Fig. 1a.

783 The respective surface fraction of  $\theta$ -fiber grains,  $\gamma$ -fiber grains and “other” grains for each  
784 EBSD map are detailed quantitatively in Fig. A.2. Results confirm the preponderance of the  
785  $\gamma$ -fiber at the center of the initial sheet with higher statistical relevance but show also that its  
786 fraction varies between 70% and 80% depending on the location of EBSD map. White  
787 rectangles on the EBSD map #1 (denoted as maps #1a and #1b) represent the size of EBSD  
788 maps performed for intergranular analysis in the present study. It appears clearly that the  $\gamma$ -  
789 fiber can be much less dominant with this size of observation as presented in Fig. A.2.

790 Indeed, the intensity of  $\gamma$ -fiber is around 70% in the left EBSD map #1a whereas it is around  
791 50% in the right EBSD map #1b. These non-negligible variations of texture can explain the  
792 differences observed between the two geometries of compression samples as the samples  
793 were taken from distant regions in the initial sheet.

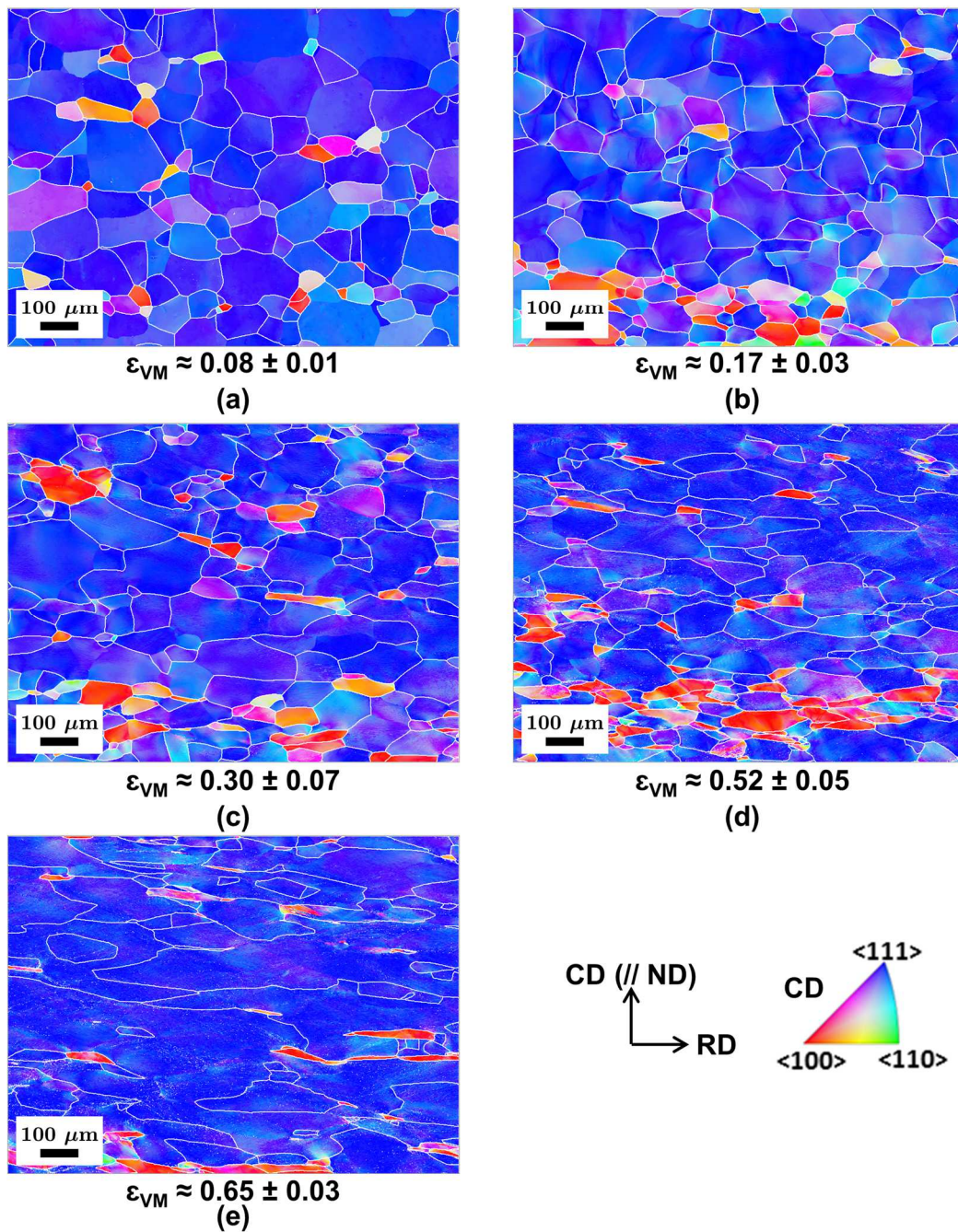


794

795 Fig. A.2. Evolution of  $\theta$ -fiber,  $\gamma$ -fiber and “other” orientations for each EBSD map of Fig. A.1.



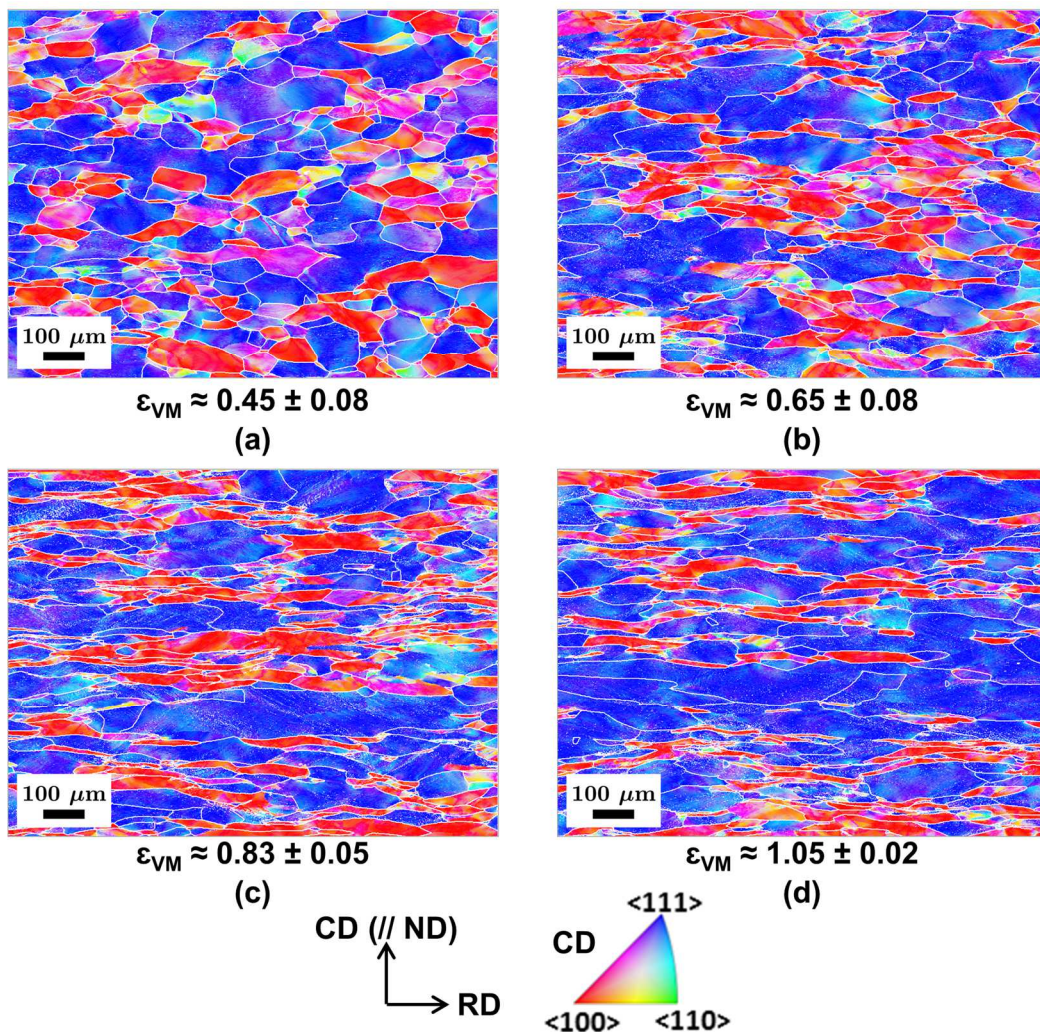
796 **Appendix B: EBSD orientation maps acquired on the geometry 1**  
 797 **double-cone compression sample**



798  
 799 Fig. B.1. EBSD orientation maps of samples deformed by compression with geometry 1 at  
 800 equivalent plastic strains of (a) 0.08, (b) 0.17, (c) 0.30, (d) 0.52 and (e) 0.65 respectively (CD  
 801 projected onto the standard triangle). Grain boundaries are plotted in white lines (10°  
 802 disorientation angle threshold).

803

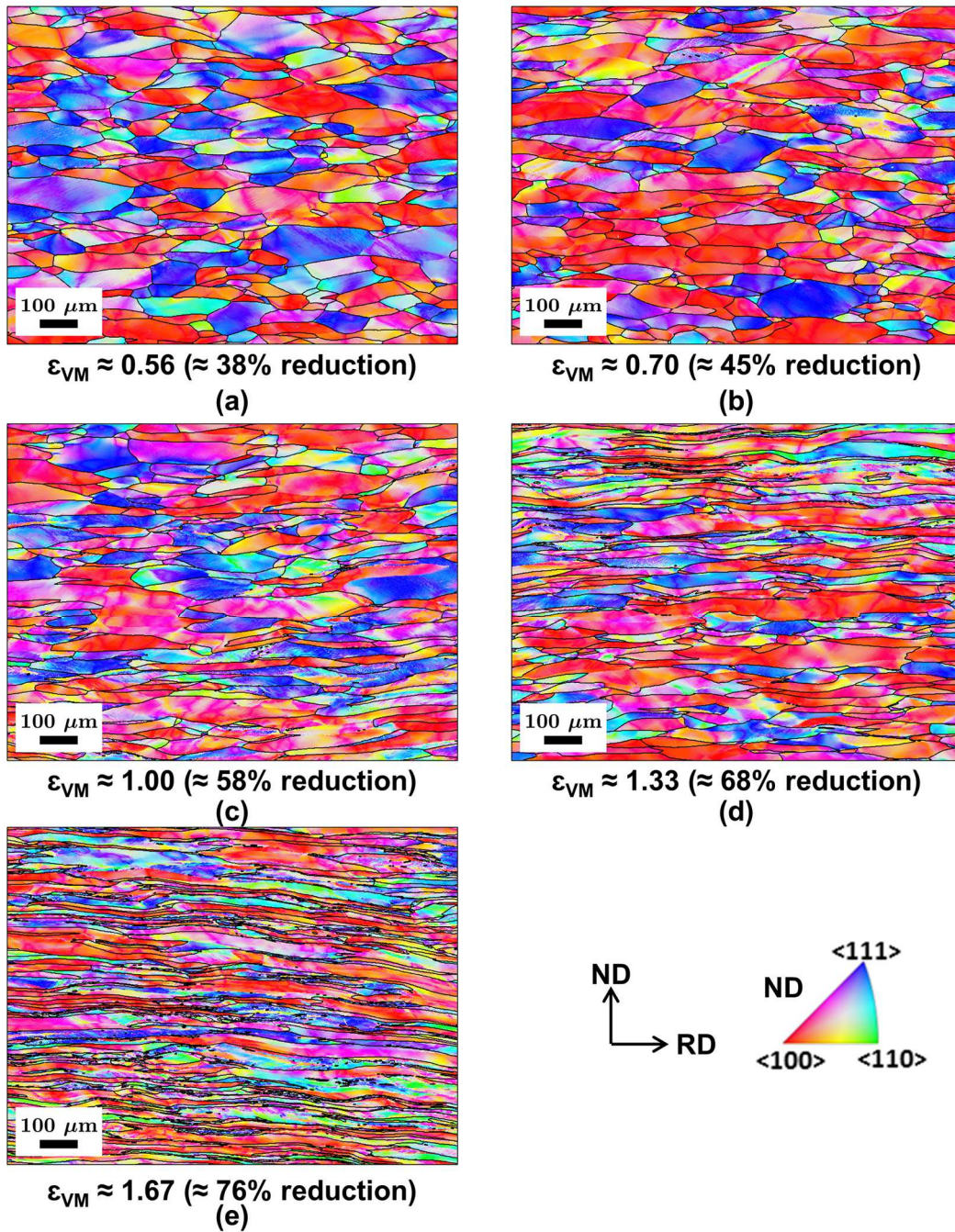
804 **Appendix C: EBSD orientation maps acquired on the geometry 2**  
 805 **double-cone compression sample**



806  
 807 Fig. C.1. EBSD orientation maps of samples deformed by compression with geometry 2 at  
 808 equivalent plastic strains of (a) 0.45, (b) 0.65, (c) 0.83, (d) 1.05 respectively (CD projected  
 809 onto the standard triangle). Grain boundaries are plotted in white lines ( $10^\circ$  disorientation  
 810 angle threshold).

811

812 **Appendix D: EBSD orientation maps acquired on rolled samples**



813  
 814 Fig. D.1. EBSD orientation maps of samples deformed by rolling at equivalent plastic strains  
 815 of (a) 0.56, (b) 0.70, (c) 1.00, (d) 1.33 and (e) 1.67 respectively (ND projected onto the  
 816 standard triangle). Grain boundaries are plotted in black lines (10° disorientation angle  
 817 threshold).