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1     **Lattice strain development in an alpha titanium alloy studied using synchrotron and**  
2   **neutron diffraction**

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14  
15     **Abstract**

16     Neutron and high-energy X-ray diffraction methods have been used to investigate  
17     deformation mechanisms (slip and twinning activities) during uniaxial tensile tests of Grade 1  
18     commercially pure titanium (Ti- $\alpha$ ). These two diffraction techniques have provided valuable  
19     and complementary information. The predictions made by an Elasto-Plastic Self-Consistent  
20     (EPSC) model, taking into account grain reorientation and stress relaxation induced by  
21     twinning activity, have been compared with the experimental data. Results clearly  
22     demonstrate that there is a good agreement between simulations and diffraction  
23     measurements. The EPSC model enables accurate predictions of: (i) the lattice strains  
24     developed in variously oriented  $\{hk.l\}$  grain families within the bulk material, (ii) the grain  
25     reorientation induced by twinning activity and (iii) the macroscopic stress-strain response.

26 Two different textured products were studied in order to evaluate the influence of texture on  
27 the mesoscopic and macroscopic responses of Ti- $\alpha$  alloy.

28 **Keywords:** titanium alloys, strain measurements, plasticity, modelling, anisotropy, diffraction

## 29 1. Introduction

30 Due to their high specific strength, good corrosion resistance and biocompatibility, titanium  
31 and its alloys are of the utmost importance for aeronautic applications and are also widely  
32 used in naval and biomedical industries [1–3]. Hexagonal Close Packed (HCP) metals present  
33 a low crystalline symmetry as compared to cubic materials. They are then governed by a  
34 heterogeneous plastic deformation, and the way it is accommodated is still not well  
35 understood. At room temperature, it is well experimentally established that prismatic slip  
36  $\{10.0\}\langle 11.0 \rangle$  is the easiest deformation mode to be activated in HCP titanium. Pyramidal  
37  $\langle c + a \rangle$  slip  $\{10.1\}\langle 11.3 \rangle$ , pyramidal  $\langle a \rangle$  slip  $\{10.1\}\langle 11.0 \rangle$  and basal slip  $\{00.2\}\langle 11.0 \rangle$  are  
38 generally presented as secondary slip modes [4,5] that are more difficult to activate. In  
39 particular,  $\{10.1\}\langle 11.3 \rangle$  slip produces plastic deformation with a component out of the basal  
40 plane (i.e. along the  $\vec{c}$ -axis). Deformation along  $\vec{c}$ -axis can also be accommodated by twinning  
41 modes  $\{10.2\}\langle 10.\bar{1} \rangle$  or  $\{11.2\}\langle 11.\bar{3} \rangle$  that induce a reorientation of the crystal lattice [4,6,7].  
42 The relative activity of these multiple deformation mechanisms strongly depends on the  
43 chemical composition, grain size, morphological and crystallographic textures, straining  
44 direction and strain rate. Understanding how the different deformation mechanisms operate  
45 during plastic deformation of titanium alloys require thus further investigations. A deeper  
46 knowledge of the mechanical behavior of HCP materials during plastic deformation calls for  
47 the use of advanced techniques such as Time-Of-Flight (TOF) neutron or High Energy  
48 synchrotron X-Ray Diffraction (HEXRD) enabling to monitor the evolution of internal elastic  
49 strains. With a high spatial resolution, X-ray synchrotron radiation allows a very small gauge

50 volume ( $200\ \mu\text{m} \times 200\ \mu\text{m}$  spot size for the present study) as compared to the neutron  
51 diffraction one ( $4\ \text{mm} \times 4\ \text{mm}$  spot size for the present study), but offers fast rate data  
52 acquisition (few seconds for synchrotron radiation and around 40 minutes for neutron  
53 diffraction in this work). This technique enables to measure online strain distributions and *in*  
54 *situ* during a continuous mechanical loading. In contrast, for materials characterized by a  
55 substantial creep behavior such as titanium alloys, the samples investigated by neutron  
56 techniques have to be unloaded once the desired macroscopic strain has been reached.  
57 Analyses relate thus only to the residual lattice strains remaining after a given increment of  
58 plastic deformation [8–10]. These two complementary non-destructive methods have been  
59 adopted in this work to measure the change in the  $\{hk.l\}$  d-spacing during tensile loadings in  
60 order to characterize the internal elastic strains (or intergranular strains) distribution.  
61 Experimental results can be explained and interpreted using a polycrystalline modeling  
62 approach like the Elasto-Plastic Self-Consistent (EPSC) model [11–15]. This  
63 micromechanical modelling is a helpful technique to characterize the mechanical state of  
64 polycrystalline materials at the grain level and to understand the role played by plastic  
65 anisotropy, slip-slip (and slip-twin) competitions in the macroscopic behavior. For example,  
66 Warwick et al. [8] have examined a textured Grade 1 Commercially Pure titanium (CP Ti) by  
67 coupling neutron diffraction to self-consistent modelling. A good agreement was found  
68 between neutron diffraction and the simulated results. The authors found that twinning  
69 produces a greater hardening than slip and that the residual lattice strains developed in the  
70  $\{00.2\}$  grains are higher than in other orientations for both tensile and compression straining.  
71 A similar finding has been achieved by Gloaguen et al. [9], who analyzed the deformation  
72 mechanisms involved in the plasticity of Grade 2 CP Ti during compressive loadings. The  
73 residual lattice strain of different  $\{hk.l\}$  orientations was tracked in both parallel and  
74 transverse directions to the straining axis and the diffraction data were investigated to

75 highlight the role of tensile twin  $\{10.2\}$  in the  $\alpha$ -titanium alloy. Cho et al. [10] used a similar  
76 methodology to characterize the intergranular residual strains accumulation in a near  $\alpha$ -  
77 titanium alloy and a Ti-834 alloy during tensile tests. It has been found that it was possible to  
78 successfully reproduce the trends and magnitudes of the observed microstrains in both loading  
79 and transverse directions. The plastic behavior of a two phase titanium alloy, Ti-6Al-4V, has  
80 also been studied [16] thanks to this coupled approach. The X-ray synchrotron diffraction has  
81 likewise been used with EPSC modelling by Stapleton et al. [17] to analyze the evolution of  
82 internal lattice strains in a Ti-6Al-4V alloy during *in situ* tensile tests. A similar study has  
83 been carried out in a near  $\beta$ -titanium alloy [18]. The main advantage of high energy X-rays is  
84 the possibility to have short measurements during a continuous mechanical test. The number  
85 of diffracting grains is nevertheless smaller as compared to neutron diffraction experiments  
86 due to the smaller gauge volume (respectively  $200\ \mu\text{m} \times 200\ \mu\text{m} \times 2\ \text{mm}$  and  $4\ \text{mm} \times 4\ \text{mm} \times$   
87  $4\ \text{mm}$ ). Therefore, the lattice strains determined with synchrotron X-ray radiation are less  
88 representative due to a smaller number of crystallites contributing to the diffracting volume.  
89 The comparison with a homogenization approach is then more problematic [14].

90 The purpose of the work presented in this paper is to understand the internal strain  
91 development of a commercially pure titanium under uniaxial tensile loadings. Experimental  
92 observations are coupled with the predictions of the EPSC model in order to explain the  
93 plastic behavior of Ti- $\alpha$  and to identify the slip systems and twinning modes responsible for  
94 the plastic activity within the material. One of the objectives of this study is to evaluate the  
95 influence of crystallographic texture and twin activity on the mechanical response. An EPSC  
96 model, taking into account grain reorientation and stress relaxation induced by twinning  
97 activity, has been developed for this study. Two different textured samples (a unidirectional  
98 rolled plate and a forged bar) for the same alloy were studied in order to evaluate the  
99 influence of texture on the mesoscopic and macroscopic responses of a Grade 1 CP Ti- $\alpha$  alloy.

100 A comparison between these two techniques and the experimental internal (X-ray) and  
101 residual (neutron) strain values obtained with an EPSC model has been made to analyze the  
102 potential difference.

## 103 2. Experimental methods

### 104 2.1. Material

105 The investigated material is a Commercially Pure  $\alpha$ -titanium (denoted CP Ti- $\alpha$ ). Two  
106 samples, produced by two different forming processes, are studied: a unidirectional rolled  
107 plate and a forged bar. The samples are fully hexagonal compact phased at room temperature  
108 and their chemical compositions are given in **Table 1**. The tensile specimens have different  
109 geometries (see **Fig. 1**).

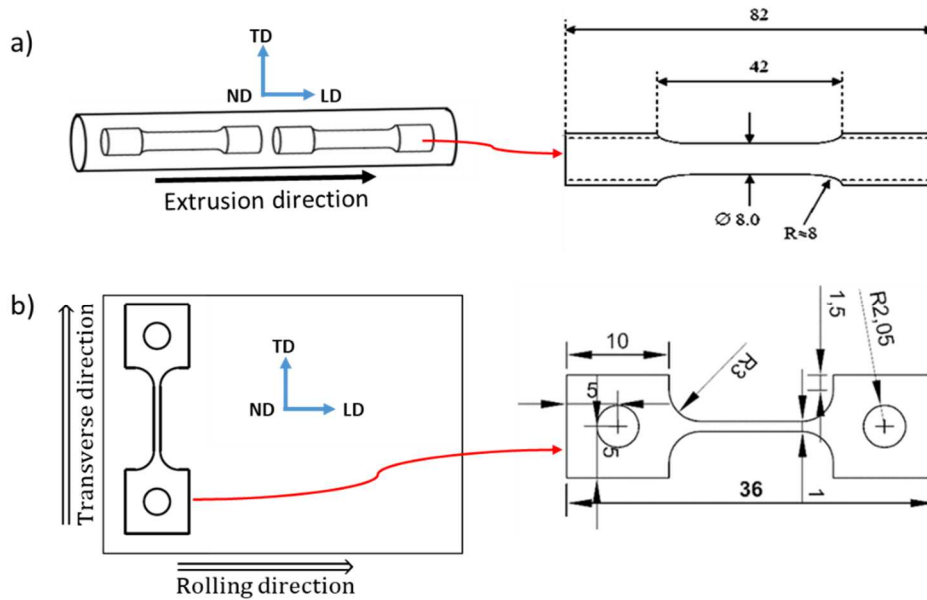
110 **Table 1:** Chemical composition data (wt.%) of the materials

Specimen	C	N	O	H	Fe	Ti
N-Sample	0.07	0.008	0.12	0.0013	-	Balance
S-Sample	0.012	0.0078	0.137	0.001	0.164	Balance

111  
112 Cylindrical specimens (denoted below as N-sample) with 8 mm diameter and 42 mm gauge  
113 length were cut along the extrusion direction (represented by the Longitudinal Direction: LD,  
114 **Fig. 1.a**) for neutron diffraction experiments. This geometry was adopted in order to access to  
115 a sufficiently large gauge volume. However, *in situ* synchrotron experiment specimens,  
116 denoted as S-samples, have been machined along the Transverse Direction (TD) of the rolled  
117 plate, as shown on **Fig. 1.b**. The sample thickness was small enough (2 mm) to enable  
118 transmission measurements [19]. The metallographic characterization has revealed that the

119 grains are generally equiaxed with an average grain size of 40 and 45  $\mu\text{m}$  in S-samples and N-  
120 samples, respectively.

121



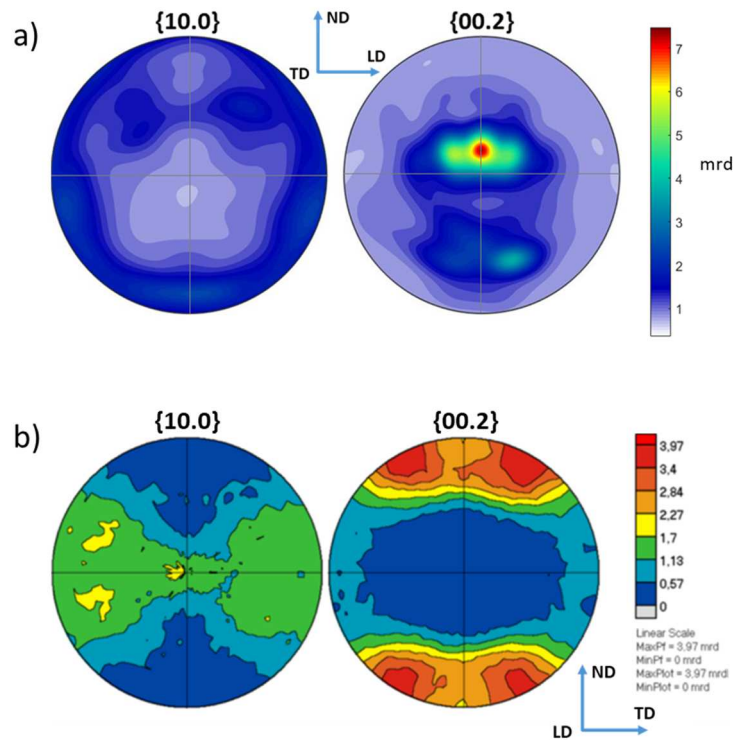
122

123 **Fig. 1:** Schematic view showing dimensions (in mm) of the samples used in a) neutron and b)  
124 synchrotron experiments. LD: Longitudinal (loading) Direction, TD: Transverse Direction,  
125 ND: Normal Direction

126 N-sample texture measurements were performed on the GEM beamline at the ISIS neutron  
127 spallation source (United Kingdom). Information about the instrument and the measurement  
128 technique can be found in [20,21]. Measurements of the initial texture (Fig. 2.a) have revealed  
129 that the samples have a strong asymmetric texture in the TD-ND plane, perpendicular to the  
130 loading axis (LD). This texture is consistent with the analyses made on extruded hexagonal  
131 polycrystalline samples found in literature [8,9,22].

132 The S-sample initial texture, shown in (Fig. 2.b), was measured using the neutron diffraction  
133 on the 6T1 diffractometer from the French national Orpheus nuclear reactor (at Léon Brillouin  
134 Laboratory, Saclay, Gif-sur-Yvette, France). As revealed in Fig. 2.b, basal poles are tilted +

135 30° away from the Normal Direction axis (corresponding to the plate normal direction)  
136 toward the Transverse Direction. The observed texture is typical of rolled titanium plates  
137 followed by recrystallization.



138

139 **Fig. 2:** Initial crystallographic textures of the studied specimens a) N-sample and b) S-sample

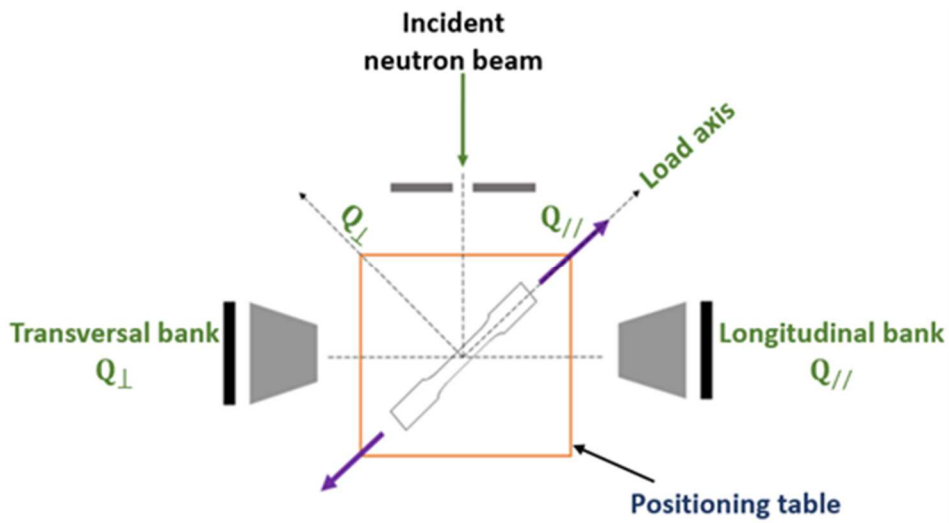
140

## 141 2.2. Neutron diffraction measurements

142 Tensile tests were performed at room temperature using the ENGIN-X neutron diffractometer  
143 (Fig. 3) at the ISIS spallation neutron source. The measurement steps were applied by a 50 kN  
144 Instron testing machine with a strain rate of  $10^{-5} \text{ s}^{-1}$  up to 6 % of macroscopic strain. The  
145 specimen was subjected to a displacement controlled regime. The incident slits and radial  
146 collimators defined a scattering volume of  $4 \times 4 \times 4 \text{ mm}^3$  with a counting time of 43 minutes  
147 per measurement. The macroscopic strain was determined using an extensometer that spanned  
148 the irradiated region. The sample was first deformed to the desired macroscopic strain and



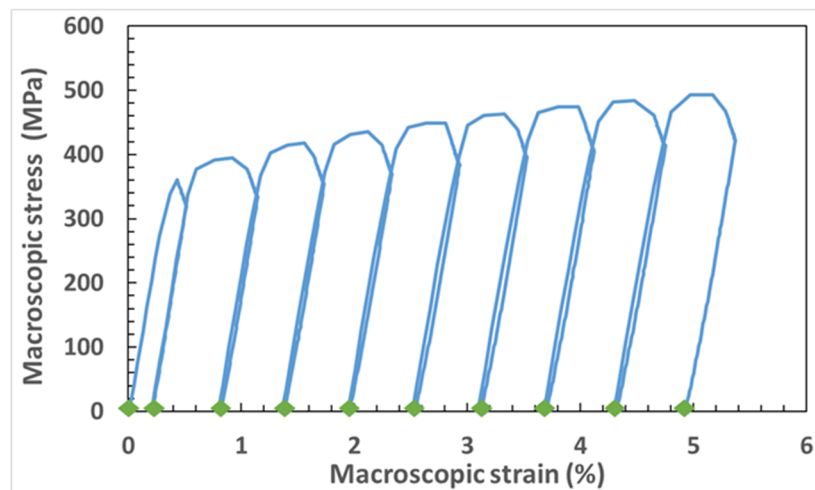
149 then immediately unloaded, as shown in Fig. 4, to avoid any strain relaxation due to the creep  
150 behavior.



151

152 **Fig. 3:** Schematic top-view of the set-up used on the neutron diffraction instrument ENGIN-X

153



154

155 **Fig. 4:** Measured macroscopic stress– strain curve obtained with ENGIN-X. The green points  
156 indicate the macroscopic strain during neutron measurements after unloading, i.e. at zero  
157 applied load.

158 Successive steps have been achieved at room temperature, in the plastic regime, studying the  
159 remaining residual strains resulting from a given increment of plastic deformation at zero

160 applied load. The schematic view of the ENGIN-X instrument (Fig. 3) shows a horizontal  
 161 loading axis positioned at 45° with respect to the incident beam. Two detector banks are set  
 162 up horizontally at ± 90° from the incident beam, enabling simultaneous measurements of  
 163 lattice strains from {hk.l} plane families with parallel (i.e. along the loading direction) and  
 164 perpendicular (i.e. along the transverse direction) directions, as regard to the loading applied  
 165 direction (Fig. 3). The Time-Of-Flight (TOF) technique enables to collect simultaneously an  
 166 entire diffraction pattern (the effective d-spacing range from 0.88 to 2.63 Å) on each detector  
 167 [23]. Single peak fitting was performed using the Open Genie code [24] with Gaussian  
 168 functions. The lattice strain  $\langle \varepsilon(hk.l, \varphi, \psi) \rangle_{V_d}$  of a grain group having a common {hk.l} plane-  
 169 normal parallel to the diffraction vector  $\vec{Q}$  (defined by azimuthal and inclination angles,  
 170 respectively  $\varphi$  and  $\psi$ ) can be calculated by the following formula:

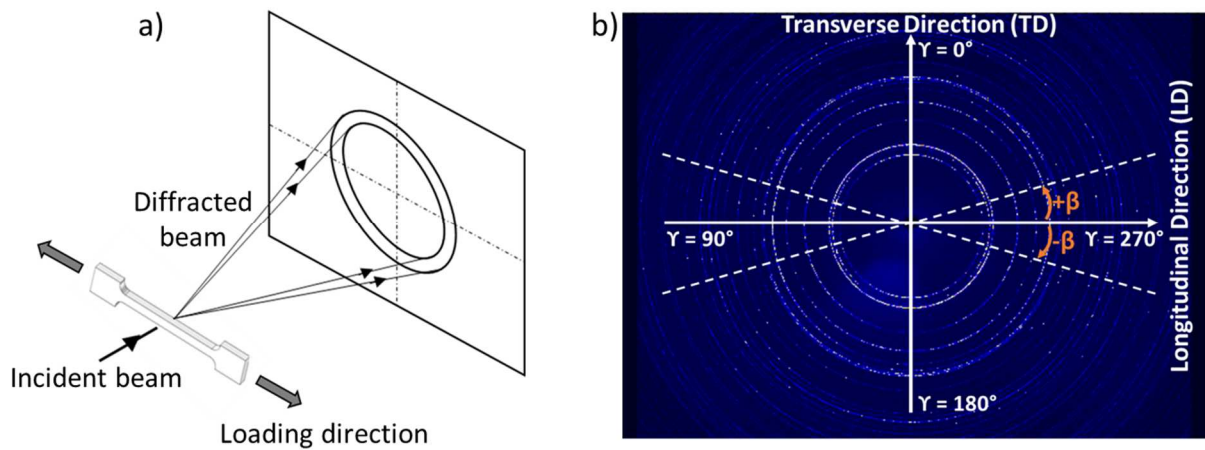
$$\langle \varepsilon(hk.l, \varphi, \psi) \rangle_{V_d} = \ln \left( \frac{\langle d(hk.l, \varphi, \psi) \rangle_{V_d}}{d_0(hk.l)} \right) \quad Eq. 1$$

171 The initial lattice parameter  $d_0(hk.l)$ , measured before deformation of the material, is used as  
 172 the reference point for this calculation.  $\langle X \rangle_{V_d}$  ( $V_d$ : diffracting volume,  $X = \varepsilon(hk.l, \varphi, \psi)$  or  
 173  $d(hk.l, \varphi, \psi)$ ) stands for the averaging over the diffracting grains for the considered {hk.l}  
 174 reflection.  $\langle d(hk.l, \varphi, \psi) \rangle_{V_d}$  is the measured lattice spacing and resulting lattice strain values  
 175 are given as micro-strain (denote  $\mu\varepsilon$ , i.e. units of  $10^{-6}$ ). In the remainder of the paper, // and  $\perp$   
 176 symbols respectively refer to the lattice strain in the parallel and perpendicular directions to  
 177 the stress axis (corresponding to the extrusion direction).

### 178 2.3. Synchrotron X-ray measurements

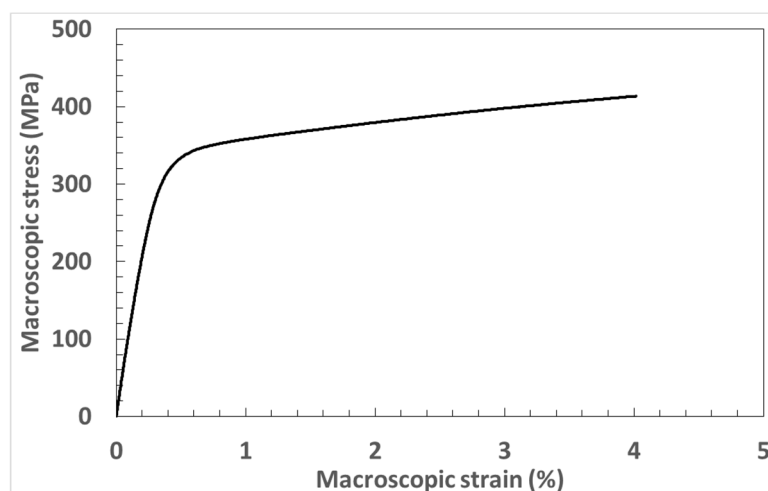
179 ID15B beamline at the ESRF (the European Synchrotron Radiation Facility, Grenoble,  
 180 France) has been used to perform *in situ* tensile tests in a transmission mode, with a  
 181 monochromatic X-ray radiation of 87 keV ( $\lambda = 0.1425$  Å wavelength). The configuration of

182 the experimental setup is shown in Fig. 5.a. The tests were performed on an Instron 8800  
183 electrothermomechanical test machine with a maximum load capacity of 3 kN under a  
184 constant strain rate  $4 \cdot 10^{-4} \text{ s}^{-1}$  up to 4 % macroscopic strain. The load was applied  
185 perpendicular to the rolling direction (Fig. 1.b). Fig. 6 shows the macroscopic curve obtained  
186 on ID15B beamline.



188 **Fig. 5:** a) Experimental setup showing S-sample positioning, b) example of image recorded  
189 on the 2D detector with  $\gamma$  and  $\beta$  angle definitions

190



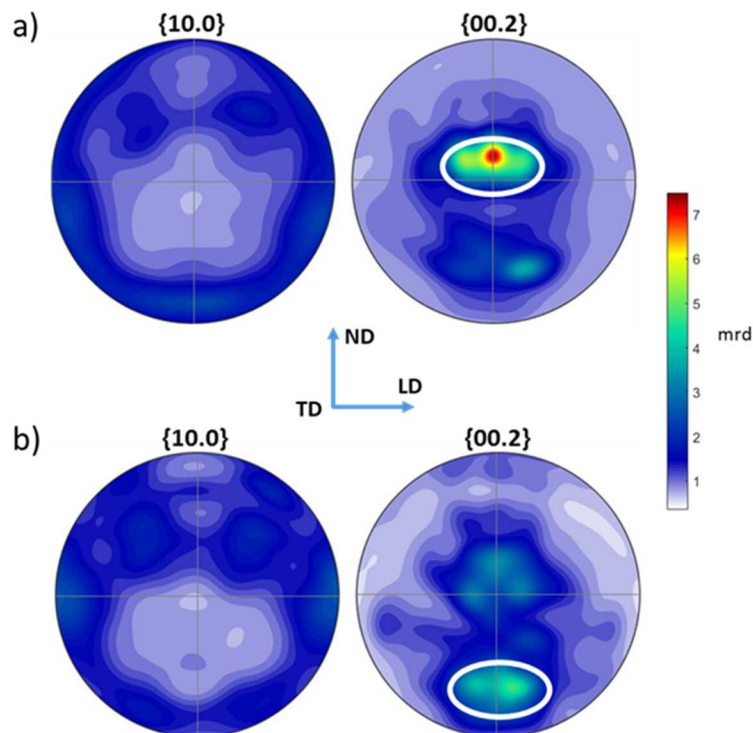
192 **Fig. 6:** Measured macroscopic stress – strain curve obtained on the ID15B beamline.

193 The diffraction patterns (example presented in Fig. 5.b), have been recorded using a 2D  
194 Pixium 4700 flat-panel detector [25], positioned in agreement with the targeted {hk.l}  
195 diffracting planes and signal optimization. Each ring recorded with the 2D detector  
196 corresponds to a {hk.l} crystallographic family plane. Regarding high  $\alpha$ -Ti creep rates, high  
197 speed data acquisition is mandatory for online measurements (2s counting time every 7s).  
198 Considering the sample dimensions (1 mm  $\times$  2 mm) and the beam size (200  $\mu$ m  $\times$  200  $\mu$ m),  
199 the irradiated volume is 0.2  $\times$  0.2  $\times$  2 mm<sup>3</sup>. It has to be underlined that this latter volume is  
200 much smaller than the gauge volume obtained in neutron diffraction experiments (4  $\times$  4  $\times$  4  
201 mm<sup>3</sup>). The recorded images were processed using the FIT2D pattern analysis software [26],  
202 which integrates the two-dimensional images (Fig. 5.b) into one-dimensional spectra  
203 (intensity  $I(\gamma, hk.l)$  vs. scattering angle  $\langle 2\theta(hk.l) \rangle_{V_d}$ ). ( $\langle d(hk.l) \rangle_{V_d}$ ,  $I(\gamma, hk.l)$ ) couples have  
204 been calculated integrating the 2D patterns over a given  $\pm \beta$  range for the two directions :  
205 longitudinal (i.e. LD, corresponding to the symmetrical azimuth angles  $\gamma = 90$  and  $270^\circ$ ) and  
206 transverse (i.e. TD, corresponding to  $\gamma = 0$  and  $180^\circ$ ). The integration range over the ring  
207 (defined by  $\pm \beta$ ) has to be reasonably large for a statistically representative behavior of the  
208 material according to the considered orientation i.e. sufficient amount of the diffractive  
209 crystallites contributing to the signal. In addition, the  $\beta$  value must be small enough to cover  
210 only crystallites oriented relatively to the considered direction. For these reasons and based on  
211 [14,19] studies, the intensities of the diffraction rings were integrated, as a compromise,  
212 within "cake shape" sectors defined by  $\beta \pm 10^\circ$ . Finally the arithmetic mean values of the d-  
213 spacings  $\langle d(hk.l) \rangle_{V_d}$  were calculated from the averaging of the symmetrically equivalent  $\gamma$   
214 and  $\gamma + 180^\circ$  angles.

215 The  $\langle 2\theta(hk.l) \rangle_{V_d}$  positions of the diffraction peaks were determined using pseudo-Voigt  
216 function fitting [27–29]. The lattice spacing  $\langle d(hk.l) \rangle_{V_d}$  was then calculated from the peak  
217 positions using the Bragg's law [30].

#### 218 2.4. Twinning activity in both N-sample and S-sample

219 Fig. 7.a shows the initial N-sample texture measured on the GEM beamline. The specimen  
220 has a strong asymmetric texture in the TD-ND plane with the majority of grains with their  
221 basal normal close to the transverse direction and perpendicular to the loading axis. After 6 %  
222 of macroscopic deformation, a reorientation of the {00.2} poles was observed (presented by a  
223 white framed area in Fig. 7.b). This can be associated with twinning deformation. Actually,  
224 the activation of compression twinning  $\{11.2\}\{11.\bar{3}\}$  produces a reorientation of the basal  
225 poles of approximately  $65^\circ$  [6,31].



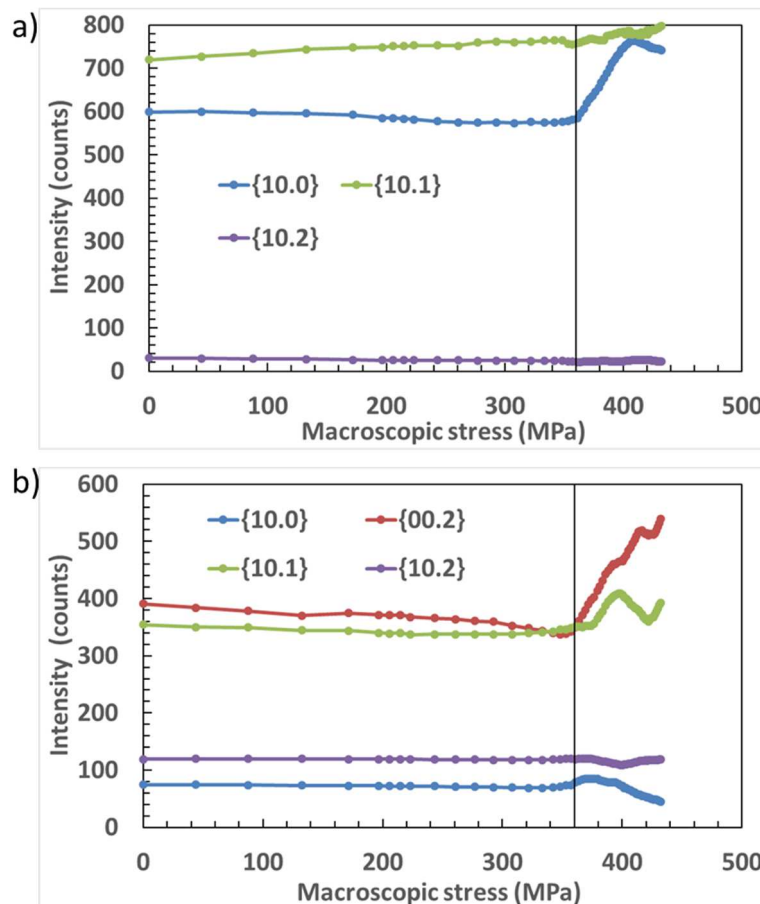
226

227 **Fig. 7:** Experimental {10.0} and {00.2} pole figures of the a) as-received N-sample and b)  
228 after the tensile loading up to 6 %. Pole figures (PFs) show the ND–LD plane, the center of  
229 the poles being the TD.

230 Tensile loading along the longitudinal (or extrusion) direction, with the  $\vec{c}$ -axis close to the  
231 transverse direction (**Fig. 7.a**), reorients the  $\vec{c}$ -axis of the grains undergoing twinning  
232 deformation. The twinned area fraction can be quantitatively calculated focusing on the  
233 difference between {00.2} pole figures of both as-received and deformed samples. Based on  
234 the work of Brown et al. [32] and integrating over the white framed area in **Fig. 7.a**, the total  
235 twin volume fraction after 6 % applied macroscopic strain is about 13 %.

236 Contrary to the previous analysis, crystallographic texture of the S-sample could not be  
237 measured in synchrotron experiments after the tensile test. Thus, analysis has focused on the  
238 evolution of the intensity measured during *in situ* straining as shown in **Fig. 8**. Gharghoury et  
239 al. [33] were the first to show that severe intensity variations during plastic deformation can  
240 be reliably attributed to twinning activity. Hence, Muránsky et al. [12], Brown et al. [32] and  
241 Gharghoury et al. [33] highlighted a direct correlation between the intensity evolution of  
242 {10.0} and {00.2} diffraction peaks, measured with detectors placed at 90° from each other  
243 and the tensile twinning {10.2}<10. $\bar{1}$ > that reorients the twin grains by 90° [31]. Our study  
244 confirms findings of these authors: a dramatic increase in {10.0} intensity in the loading  
245 direction (LD) is observed above around 365 MPa macroscopic stress (**Fig. 8.a**). At the same  
246 time, in the transverse direction (TD), the intensity of the {10.0}-orientated grains decreases,  
247 while the {00.2} peak intensity increases significantly (**Fig. 8.b**). This reflects a change in the  
248 grain volume fraction that diffracts in the TD and LD. Owing to the texture of the as-received  
249 S-sample, the changes of {00.2} and {10.0} peak intensities in these two directions are due to  
250 tensile twinning {10.2}<10. $\bar{1}$ > activity.

251 The evolution of the intensity of others reflections such as {10.1} in TD (Fig. 8.b) could  
 252 naturally also be related to other twinning mode activity but it is difficult to discern which one  
 253 only in the light of the information given by present synchrotron X-ray diffraction  
 254 experiments.



255

256 **Fig. 8:** Evolution of the diffraction peak intensities (Miller indices indicated in the plot)  
 257 obtained from *in situ* synchrotron X-ray diffraction measurements in the a) loading (LD) and  
 258 transverse (TD) directions

### 259 3. Twinning scheme for Elasto-Plastic Self-Consistent model

260 The one-site EPSC approach applied in this work incorporate the single crystal plasticity  
 261 model derived by the pioneering works of Kröner [34,35] and Hill [36]. It adopts a weighted  
 262 population of grains approach which exploits the experimentally measured crystallographic

263 textures as inputs. Each grain is assumed to have the form of an ellipsoidal inclusion  
 264 embedded in a homogeneous effective medium with anisotropic properties representative of  
 265 the textured polycrystalline aggregate. The model has been extensively described in [9,15,37],  
 266 therefore only a brief description will be given here. More information concerning the  
 267 approach used can also be found in [38–41]. This model enables to incorporate the measured  
 268 experimental results as well as to determine the strain hardening parameters, the related  
 269 Critical Resolved Shear Stress (CRSS) and the mechanical strain/stress state at the  
 270 mesoscopic level, for the diffracting volume and during the mechanical tests.

271 The slip rate  $\dot{\gamma}^g$  on a slip system  $g$  is linked to the resolved shear stress rate  $\dot{\tau}^g$  [40,41] by:

$$\dot{\gamma}^g = T^g(\tau^g, \tau_c^g, \dot{\tau}^g) \dot{\tau}^g \quad \text{Eq. 2}$$

272 where  $\tau^g$  is the resolved shear stress and  $\tau_c^g$ , the critical resolved shear stress.  $T^g$  is the  
 273 deformation mechanisms management function and is expressed by:

$$T^g = \frac{1}{H^{gg}} \left( \frac{1}{2} (1 + \tanh(\tau^g)) \right) \left( \frac{1}{2} \left( 1 + \tanh \left( k \left( \frac{\tau^g}{\tau_c^g} - 1 \right) \right) \right) \right) \left( \frac{1}{2} (1 + \tanh(\dot{\tau}^g)) \right) \quad \text{Eq. 3}$$

274 where  $H^{gg}$  is the self-hardening coefficient and  $k$ , a numerical parameter. The hyperbolic  
 275 tangent function was used because it enables the mechanical and hardening behaviors,  
 276 observed in practice, to be reproduced [40,41].

277 The hardening matrix  $H^{gr}$  is introduced to describe the CRSS rate evolution in a system  $g$ , as  
 278 a function of the plastic slip (or twinning) on the other deformation systems  $r$ :

$$\dot{\tau}_c^g = \sum_r H^{gr} \dot{\gamma}^r \quad \text{Eq. 4}$$

279 where  $\dot{\tau}_c^g$  is the CRSS rate. Due to a lack of relevant experimental data on latent hardening  
 280 nature, especially in titanium alloys, the interactions between the different deformation modes  
 281 are described by a simple law. In the present work, the hardening matrix is described by the  
 282 following equation [39]:



$$H^{gr} = q.H^{gs}$$

Eq. 5

283  $H^{gs}$  is the self-hardening coefficient and  $H^{gr}$  corresponds to the latent hardening when  $g$  slip  
284 system is different from  $r$ . The  $q$  factor defines the degree of latent hardening.

285

286 In the present study, twinning deformation is treated as a directional slip mechanism [42–44].

287 It is characterized by the normal to the twinning plane  $n^t$  and the twinning direction  $m^t$ .

288 When a twinning system becomes active, a part of the parent grain is reoriented and stress  
289 relaxation is introduced. In order to take into account these phenomena, different algorithms

290 have been proposed. Van Houtte [45] was the first to propose a “Monte Carlo” approach

291 consisting of randomly sampling and reorienting full grains. A scheme of such an approach

292 has been proposed by Tomé et al. [46]: reorienting the grain according to the most active

293 twinning system, named Predominant Twin Reorientation (PTR) formulation. Lebensohn and

294 Tomé [47] have proposed another one called a Volume Fraction Transfer (VFT) formulation.

295 The Euler space is partitioned in cells and, instead of keeping the grain volume fraction

296 constant while changing its orientation, as done in PTR, the volume fraction assigned to each

297 cell evolves with deformation. This last approach enables to predict the crystallographic

298 texture evolution but it has the disadvantage of losing the grain identity and it does not allow

299 to implement a realistic hardening scheme.

300 The scheme proposed in this paper, called Modified Predominant Twin Reorientation (M-

301 PTR), is based on Tomé et al. [46] and Clausen et al. [48] assumptions.

### 302 3.1 Modified Predominant Twin Reorientation (M-PTR)

303 The M-PTR scheme accounts for: (i) the stress relaxation associated with the twin formation

304 and, (ii) the twinning fraction effect on texture evolution. At each imposed strain increment,

305 the philosophy of the PTR scheme is adopted. The accumulated shear of each twin system in

306 each grain is recorded and the Predominant Twin System (PTS) is identified as the one that  
 307 involves the maximum volume fraction  $f_{cum}^t$  (Eq. 6):

$$f_{cum}^t = \sum \frac{\dot{\gamma}^t}{S^t} \quad Eq. 6$$

308 Here,  $\dot{\gamma}^t$  represents the shear due to the twin system  $t$  and  $S^t$  is the characteristic twinning  
 309 shear.

310 Once the predominant twin system is identified, the total fraction  $f_{cum}^{gr}$  accumulated in each  
 311 grain is given by the sum over all  $N^t$  active twin systems:

$$f_{cum}^{gr} = \sum_{t=1}^{N^t} f_{cum}^t \quad Eq. 7$$

312 In the EPSC model, a threshold fraction  $f_{limit}$  is introduced in Eq. 8. The idea behind this  
 313 parameter is to take into account the twinning nucleation mechanism and the initial growth  
 314 process in the parent grain. More information about these phenomena can be found in [49–  
 315 52]. When the volume fraction  $f_{cum}^{gr}$  in a parent grain reaches the threshold  $f_{limit}$ , that fulfills  
 316 the following equation (Eq. 8), the PTS system is selected and the corresponding grain is fully  
 317 reoriented.

$$f_{cum}^{gr} \geq f_{limit} \quad Eq. 8$$

318  $f_{limit}$  is the model parameter that will be adjusted. Based on Tomé et al. [46], Tomé and  
 319 Lebensohn [53] and Wronski et al [54] studies, the threshold value  $f_{limit}$  is taken between 5  
 320 and 50 %. If  $\Omega^m$  is the parent grain orientation, the twin orientation (child)  $\Omega^{tw}$  will be  
 321 determined by the relation (Eq. 9) [55,56]:

$$\Omega^{tw} = R^{tw} \Omega^m \quad Eq. 9$$

322 The lattice rotation tensor  $R^{tw}$  is given by:

$$R^{tw} = 2n^t \otimes n^t - I \quad Eq. 10$$

323 where  $n^t$  is the twinning plane normal and  $I$  is the unit tensor.

324 Due to the fact that the twinning deformation is a secondary deformation mechanism in our  
325 study (see [section 2](#)), the nucleated twin is treated as a new independent grain and the new  
326 orientation is kept constant until the end of the simulation.

### 327 *3.1.1 Twin variant selection criterion*

328 Experimental studies have shown that each active twin system has six possible variants.  
329 Therefore, it is important to define a criterion allowing for the selection of the most  
330 appropriate twinning variant. Electron Backscatter Diffraction (EBSD) statistical analyses on  
331 zirconium [\[50\]](#) showed that {10.2} twinning variants with the highest Schmid factors were the  
332 most active (about 56-60 % activation probability). Similar dependencies were observed for  
333 {10.2} and {11.2} twinning systems during tensile or compressive loading of commercially  
334 pure titanium in [\[54,57,58\]](#). However, it has been noticed that variants with lower Schmid  
335 factors may also contribute to plastic deformation. Thereby, it is clear that the selection  
336 process is crucial since it affects the microstructure and the texture evolution of the sample.  
337 For example, Wronski et al [\[54\]](#) have used a Visco-Plastic Self-Consistent (VPSC) model  
338 with a simple Monte Carlo twin variant selection criterion to predict with accuracy the  
339 crystallographic texture evolution in titanium during tensile and compression tests. On the  
340 basis of these analyses and in order to provide a reasonable description of the selection  
341 process, one variant is picked randomly among the three with the highest twin fractions. The  
342 goal behind is to allow for the relatively less predominant variants to accommodate plastic  
343 deformation.

### 344 *3.1.2 Estimation of the stress relaxation*

345 As mentioned previously, twinning activity induces not only the twinned zone reorientation  
346 but also a stress relaxation in the parent grain [\[48,59,60\]](#). Stress relaxation has been  
347 implemented on the assumptions made in [\[48,59,60\]](#). Elastic strain and stress, originally

348 present and developed in the new formed (child) grain are estimated by applying continuity  
 349 equations for tractions and displacements across the twin boundary. The stress and strain at  
 350 the grain level are assumed to be uniform in the present model and the elastic strains within  
 351 the plane of the child twin are supposed to be compatible to the matrix on the other side of the  
 352 twin boundary as described in [48]. Furthermore, stresses acting on the twinning plane are also  
 353 assumed to be equal across the twin interface. When the newly child grain is created and  
 354 grown to an accumulated twin volume fraction  $f_{cum}^t$  (given in Eq. 6) of the parent grain, a  
 355 shear strain  $\gamma^t$  for the twin system  $t$  is calculated from the equation below:

$$\gamma^t = f_{cum}^t S^t \quad \text{Eq. 11}$$

356 The plastic shear  $\gamma^t$  generates necessarily a back-stress between the parent and child grains in  
 357 order to accommodate the plastic deformation. It is determined thanks to the Eq. 12 assuming  
 358 that the imposed plastic shear is equal and opposite to the elastic shear:

$$\varepsilon^{II0} = -R^t \gamma^t \quad \text{Eq. 12}$$

359 Here,  $\varepsilon^{II0}$  represents the elastic back-strain and  $R^t$ , the Schmid tensor for the twin system  $t$ .  
 360 According to Clausen et al. [48], parent crystal is assumed to be infinitely stiff in comparison  
 361 with the newly formed twin. It is thus not accommodating any of the plastic shear, forcing the  
 362 twin to develop an equal and opposite elastic strain. The back-stress is computed with the  
 363 following relation (Eq. 12), using the twin elastic stiffness tensor:

$$\sigma^{II0} = c^t \varepsilon^{II0} \quad \text{Eq. 13}$$

364 In the formulation applied, the parent and the twin child single-crystal elastic coefficients are  
 365 supposed to be the same. Then, the stress relaxation is introduced by correcting the current  
 366 stress with the calculated back-stress  $\sigma^{II0}$ . The sum is made over the twin systems  $t$  which  
 367 become active in the current step [48,60]:

$$\sigma^{II} = \frac{\sigma^{II} + \sum_t \sigma^{II0} f_{cum}^t}{1 - \sum_t f_{cum}^t} \quad \text{Eq. 14}$$

368 3.2 Model parameter determination

369 In order to perform the numerical simulations, the model parameters must be identified to  
 370 describe simultaneously the macroscopic stress-strain curve and the lattice strains measured on  
 371 the various {hk.l} reflections. The single crystal elastic constants used in these simulations are  
 372 shown in Table 2.

373 **Table 2:** The single crystal elastic constants (GPa) used in M-PTR model

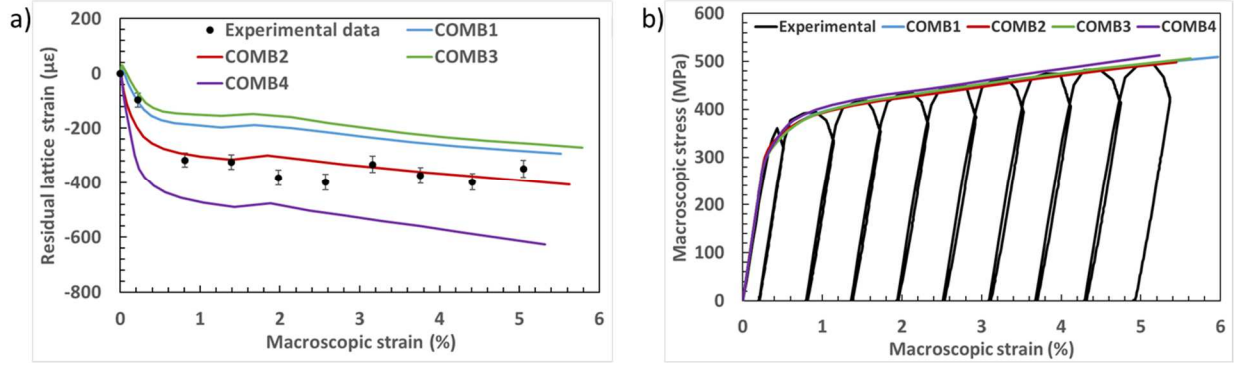
Reference	$c_{11}$	$c_{12}$	$c_{13}$	$c_{33}$	$c_{44}$	$c_{66}$
[61]	162.4	92	69	180.4	46.7	35.2

374  
 375 Experimental PFs were used to generate the Orientation Distribution Function (ODF). A set  
 376 of 2000 grain orientations weighted by the measured volume fraction has been selected from  
 377 the experimental ODF. The choice of this number is the result of a preliminary study that  
 378 allows us to determine the sufficient numbers of grains which describe correctly the  
 379 experimental data (i.e. neutron or synchrotron measurements) [9,11]. On the basis of  
 380 experimental observations in [4,7,62,63], the deformation systems considered in the  
 381 simulations are: prismatic slip (denoted P⟨a⟩), basal (B⟨a⟩), pyramidal ( $\Pi_1$ ⟨a⟩), first-order  
 382 pyramidal ( $\Pi_1$ ⟨c + a⟩) and {11.2} twinning (ctw-{11.2}) for neutron data simulations and,  
 383 prismatic slip (P⟨a⟩), basal (B⟨a⟩), pyramidal ( $\Pi_1$ ⟨a⟩), first-order pyramidal ( $\Pi_1$ ⟨c + a⟩),  
 384 {10.2} twinning (ttw-{10.2}) and {11.2} twinning (ctw-{11.2}) for X-ray synchrotron data  
 385 simulations. In the two cases, the optimal value assigned to the parameter k (see Eq. 3) is 20.  
 386 This is to ensure the stability and numerical convergence of the simulations [40,41].

387 As previously pointed out, owing to a lack of relevant data on the nature of latent hardening in  
 388 HCP titanium alloys and to be more consistent with the theoretical and experimental  
 389 information currently available, a simple and empirical law in Eq. 5 is applied to describe the  
 390 interactions between different deformation modes. The q parameter (see Eq. 5), which

391 determines the degree of latent hardening has been adjusted to provide the best accordance  
392 between the experimental and predicted results (set to a value of 1.3 for the simulations).  
393 Concerning the twinning activity, the threshold fraction  $f_{limit}$  is set to 25 % what leads to a  
394 good description of the material behavior.

395 Due to the easy activation of prismatic deformation mode  $P\langle a \rangle$ , its CRSS value  $\tau^{P\langle a \rangle}$  is  
396 directly related to the macroscopic yield strength and the elastic-plastic transition during  
397 tensile tests. The self-hardening coefficient  $H^{P\langle a \rangle}$  was determined by fitting experimental  
398 macroscopic stress-strain curves at the plasticity activity outset. These parameters are then  
399 adjusted comparing neutron and synchrotron diffraction measurements. About basal ( $B\langle a \rangle$ ),  
400 pyramidal ( $\Pi_1\langle a \rangle$ ), first-order pyramidal ( $\Pi_1\langle c + a \rangle$ ),  $\{10.2\}$  twinning ( $ttw\{-10.2\}$ ) and  
401  $\{11.2\}$  twinning ( $ctw\{-11.2\}$ ), there is less consensus on their relative role on the overall  
402 behavior. These deformation mechanisms activation sequence can be found in the literature  
403 and may differ from one type of alloy to another and / or from one author to another [64–68].  
404 The main goal here is to identify a set of parameters able to explain the results obtained at  
405 both the macroscopic scale and diffracting volume level. Several numerical simulations are  
406 performed to determine the right set of parameters, in a first step, to successively favor in turn  
407 one or several mechanisms. Fig. 9.a shows an example of comparison of the simulated results  
408 for  $\{20.1\}$  reflection with different activation sequence combinations (denoted COMB1,  
409 COMB2, COMB3 and COMB4) and the measured residual lattice strain in neutron  
410 diffraction. A qualitative analysis shows that only COMB2 sequence reproduces correctly the  
411 behavior of  $\{20.1\}$  grain groups. This illustrates clearly the influence of the choice of the  
412 number of deformation modes and their hierarchy on the mechanical behavior at the  
413 diffracting volume level, even if all these combinations describe the observed macroscopic  
414 behavior (Fig. 9.b).



415

416 **Fig. 9:** a) Residual experimental (symbols) and predicted (lines) lattice strain evolution of  
 417 {20.1} grains in loading direction versus applied stress and b) Experimental and simulated

418 macroscopic stress–strain curves during neutron diffraction experiments. COMB1:  $\mathbf{P}\langle\mathbf{a}\rangle -$

419  $\mathbf{B}\langle\mathbf{a}\rangle - \Pi_1\langle\mathbf{a}\rangle - \Pi_1\langle\mathbf{c} + \mathbf{a}\rangle - \mathbf{ctw}\{-\mathbf{11. 2}\}$ , COMB2:  $\mathbf{P}\langle\mathbf{a}\rangle - \Pi_1\langle\mathbf{a}\rangle - \mathbf{B}\langle\mathbf{a}\rangle - \Pi_1\langle\mathbf{c} + \mathbf{a}\rangle -$

420  $\mathbf{ctw}\{-\mathbf{11. 2}\}$ , COMB3:  $\mathbf{P}\langle\mathbf{a}\rangle - \mathbf{B}\langle\mathbf{a}\rangle - \Pi_1\langle\mathbf{c} + \mathbf{a}\rangle - \mathbf{ctw}\{-\mathbf{11. 2}\}$  and COMB4:  $\mathbf{P}\langle\mathbf{a}\rangle - \Pi_1\langle\mathbf{a}\rangle -$

421  $\Pi_1\langle\mathbf{c} + \mathbf{a}\rangle - \mathbf{ctw}\{-\mathbf{11. 2}\}$

422 In order to obtain the final material parameters from neutron strain measurements, a least  
 423 squares method is used, in a second step, to optimize the parameters by minimizing a function  
 424  $e^2$  defined as the sum of squares between deviations of the experimental and simulated  
 425 quantities, over all  $p$  grain groups:

$$e^2 = \sum_{i=1}^p \left( \frac{\langle \varepsilon(hk.l, \varphi, \psi) \rangle_{V_d meas}^i - \langle \varepsilon(hk.l, \varphi, \psi) \rangle_{V_d simul}^i}{u(\langle \varepsilon(hk.l, \varphi, \psi) \rangle_{V_d meas}^i)} \right)^2 \quad Eq. 15$$

426

427 where  $\langle \varepsilon(hk.l, \varphi, \psi) \rangle_{V_d meas}^i$  is the measured value,  $\langle \varepsilon(hk.l, \varphi, \psi) \rangle_{V_d simul}^i$  the simulated

428 value, and  $u(\langle \varepsilon(hk.l, \varphi, \psi) \rangle_{V_d meas}^i)$  the uncertainties of the measured lattice strains.

429 The final set of values that gives the optimum agreement between the measured and simulated

430 results are summarized in [Table 3](#).

431 **Table 3:** Optimized set of CRSS and hardening coefficients (MPa) obtained with the model.  
 432 In blue, CRSS normalized values with respect to  $\text{CRSS}^{\text{P(a)}}$ , determined with the EPSC model  
 433 and neutron diffraction results.

	Neutron diffraction		Synchrotron diffraction	
	CRSS	H <sup>gg</sup>	CRSS	H <sup>gg</sup>
<b>P</b> $\langle\mathbf{a}\rangle$	115 (1)	120	110 (1)	100
<b>B</b> $\langle\mathbf{a}\rangle$	150 (1.30)	180	150 (1.36)	150
$\Pi_1\langle\mathbf{a}\rangle$	130 (1.13)	140	120 (1.09)	120
$\Pi_1\langle\mathbf{c} + \mathbf{a}\rangle$	240 (2.09)	250	180 (1.64)	200
<b>ttw</b> -{10.2}	-	-	190 (1.73)	250
<b>ctw</b> -{11.2}	250 (2.17)	300	220 (2)	300

#### 434 4. Model results and discussion

435 Before starting the simulations, residual stresses were quantified in the as-received N-sample  
 436 and S-sample.

437 Lattice parameters  $a = 2.9509 \text{ \AA}$  and  $c = 4.6844 \text{ \AA}$  of the N-sample were found from a  
 438 Rietveld refinement [10] of the initial entire diffraction ring for measurements along the LD.

439 From these, the ideal d-spacing  $\langle d_0^{\text{Rietveld}}(hk.l) \rangle_{V_d}$  for all peaks  $\{hk.l\}$  can be calculated  
 440 from:

$$\frac{1}{\langle d_0^{\text{Rietveld}}(hk.l) \rangle_{V_d}} = \frac{4h^2 + hk + k^2}{3a^2} + \frac{l^2}{c^2} \quad \text{Eq. 16}$$

441 The difference between the predicted  $\langle d_0^{\text{Rietveld}}(hk.l) \rangle_{V_d}$  and measured d-spacing  
 442  $\langle d(hk.l, \varphi, \psi) \rangle_{V_d}$  by neutron diffraction is assumed to be due to initial residual lattice strain

443  $\varepsilon_{hk.l}^{\text{res}} \left( \varepsilon_{hk.l}^{\text{res}} = \frac{\langle d_0^{\text{Rietveld}}(hk.l) \rangle_{V_d}}{\langle d(hk.l, \varphi, \psi) \rangle_{V_d}} - 1 \right)$  in the extrusion direction (see Table 4).



444

445 **Table 4:** Initial residual lattice strains in N-sample in the extrusion direction (LD)

{hk.l}	{10.0}	{00.2}	{10.1}	{10.2}
$\epsilon_{hk.l}^{\text{res}} (\times 10^{-6})$	$91 \pm 38$	$22 \pm 50$	$-57 \pm 40$	$-62 \pm 46$
{hk.l}	{11.0}	{10.3}	{11.2}	{20.1}
$\epsilon_{hk.l}^{\text{res}} (\times 10^{-6})$	$-40 \pm 38$	$64 \pm 48$	$-55 \pm 42$	$-116 \pm 39$

446

447 Similar order of magnitude is also found along TD. This indicates that the maximum  
 448 deviation is equivalent to a strain of  $120 \times 10^{-6}$ , and thus corresponds to initial residual  
 449 stresses below 10 MPa.

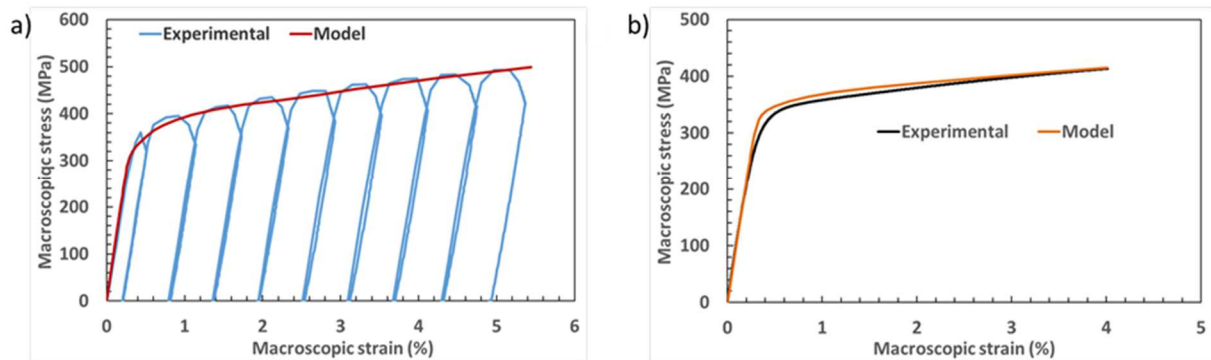
450 The as-received cold rolled sheet used in this study has already been studied in previous work  
 451 [15]. Analysis of the residual stress obtained from {30.2} and {21.3} reflections have then  
 452 shown values between  $-10 \pm 35$  MPa and  $+12 \pm 24$  MPa along the rolling direction. The stress  
 453 values along the TD are also in the same order of magnitude. Eventually, the two samples N  
 454 and S display rather low residual stress values and latter do not play, *a priori*, a significant  
 455 role on the elastoplastic behavior. For this reason, initial residual strains / stresses in the as-  
 456 received material were not accounted for in the model.

#### 457 4.1 Neutron diffraction results

458 The EPSC model developed in the section 3 is used here to simulate the mechanical behavior  
 459 of the CP Ti- $\alpha$  alloy. Experimental and predicted macroscopic responses of the N-sample  
 460 under uniaxial tensile test along the extrusion direction (or LD) is shown Fig. 10.a.

461 The plastic slope predicted by the model is in good agreement with that measured up to 5 %  
 462 macroscopic strain. The calculated value of the Young's modulus of 107 GPa is close to the

463 experimental one equal  $102 \pm 1$  GPa. This is consistent with the values commonly found in  
 464 the literature [69].



465

466 **Fig. 10:** Experimental and simulated macroscopic stress–strain curves in a) neutron and b)

467

synchrotron diffraction experiments

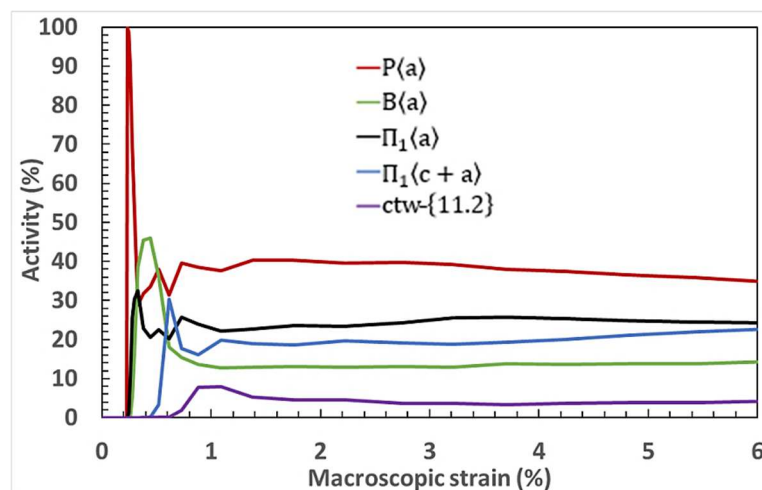
468 **Fig. 11** shows the predicted relative activity of each deformation mode considered in the

469 simulation. The experimental macroscopic yield stress is about 280 MPa corresponding to

470 0.29 % total strain. The adjusted CRSS values are consistent with the EBSD analyses of

471 Barkia et al. [67] with the following order:  $P\langle a \rangle$  -  $\Pi_1\langle a \rangle$  -  $B\langle a \rangle$  -  $\Pi_1\langle c + a \rangle$  -  $ctw\{11.2\}$ .

472



473

474 **Fig. 11:** Relative contribution of each deformation mode to the total plastic deformation as a  
475 function of deformation during neutron diffraction experiments

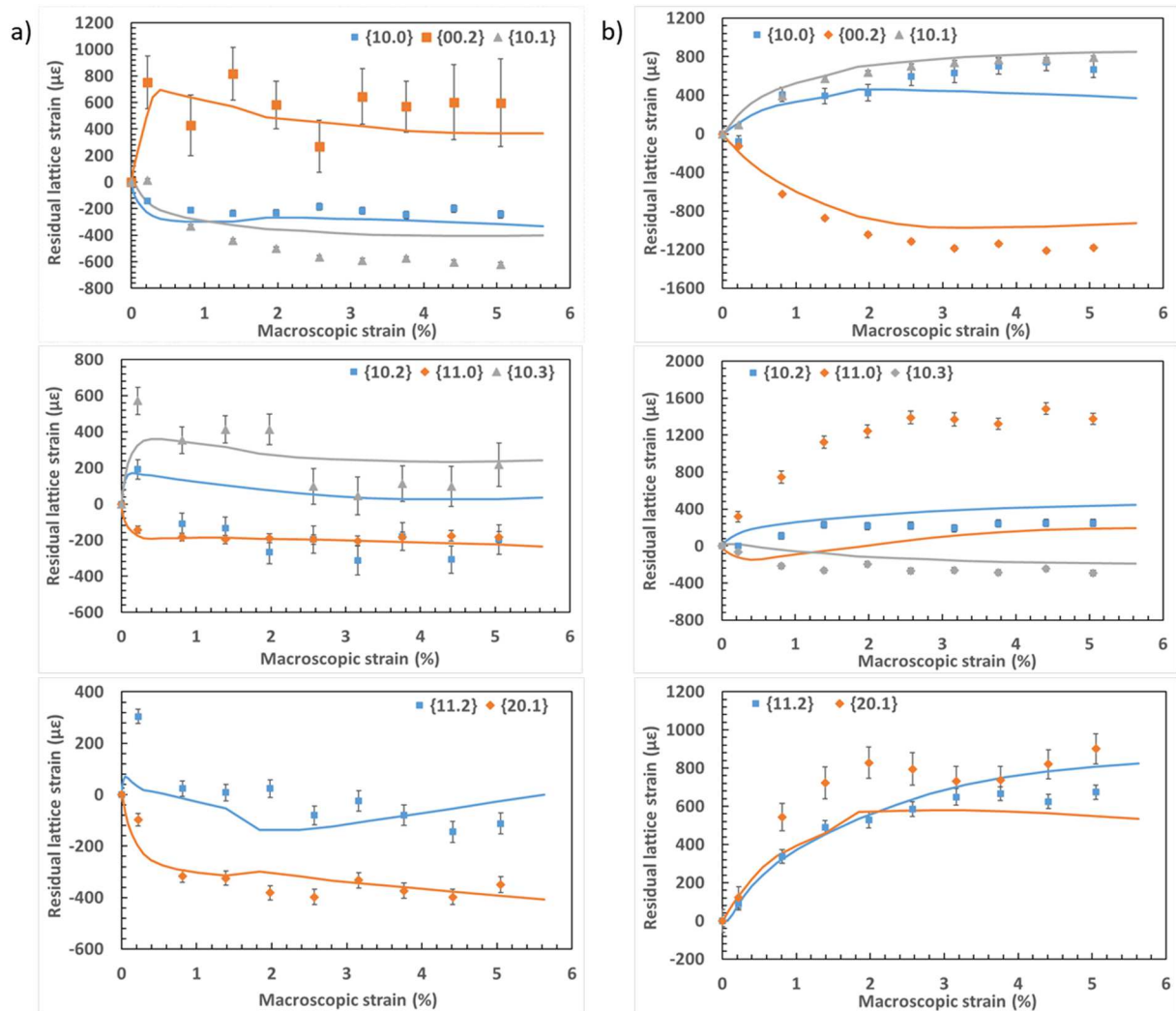
476 The main deformation mode is prismatic slip. For example, at 3 % macroscopic total strain,  
477 the proportions of activated systems are: 39 % for prismatic slip, 13 % for basal slip, 25 % for  
478 pyramidal  $\langle a \rangle$  slip, 18 % for pyramidal  $\langle c + a \rangle$  and 3.5 % for  $\{11.2\}$  twinning.

479 This activation sequence (Fig. 11) will influence the behavior of the polycrystal at the local  
480 level (i.e. at the diffracting volume level). Then, it is interesting to see its impact on the  
481 different mechanical states of  $\{hk.l\}$  grain families. To illustrate this, the evolution of residual  
482 intergranular strains is shown in Fig. 12 for both longitudinal (loading direction) and  
483 transversal directions. When a polycrystalline aggregate is subjected to a tensile test in the  
484 elastic region, the lattice strains develop linearly with the load up to the micro-yielding point.  
485 There is a significant difference in the elastic response for each  $\{hk.l\}$  grain family due to the  
486 elastic anisotropy and the range of possible crystallographic orientations. Slip activity is  
487 initiated in plasticity regime in some grains similarly orientated. One can notice the  
488 accommodation of the imposed plastic deformation and these grains undergo less loads.

489 The neutron experiments results shown in Fig. 12 clearly highlight an anisotropic mechanical  
490 behavior of  $\{hk.l\}$  groups of grains.

491 According to the simulations, plasticity initiation begins with the yielding of  $\{10.0\}_{//}$  grains  
492 at 260 MPa (corresponding to 0.24 % of total strain). These grains are favorably oriented for  
493 prismatic slip  $P\langle a \rangle$ , the lowest-CRSS mechanism, correlated with a favorable Schmid factor  
494 (i.e. 0.5) for these orientations [4,6,67]. Indeed, the grains that activate the prismatic slip first  
495 and most easily are those which have their lattice crystalline  $\vec{c}$ -axis perpendicular to the  
496 loading direction. These grains undergo plastic deformation below the macroscopic yield  
497 stress ( $R_E = 280$  MPa). It should be recalled that the outset of microplasticity does not cause

498 the macroscopic yield because slip activity is initiated in a small number of grains. As the  
 499 applied stress increases,  $\{20.1\}_{//}$  orientation begins to shed load at 300 MPa. This can be  
 500 explained by the activation of pyramidal slip  $\Pi_1\langle a \rangle$ . The  $\{10.1\}_{//}$  and  $\{10.2\}_{//}$  orientated  
 501 grains yield at 330 MPa, reflecting the high activity of the basal slip ( $\sim 45\%$  of activity  
 502 proportion).



503

504 **Fig. 12:** Residual experimental (symbols) and predicted (lines) lattice strain evolution in both  
 505 (a) longitudinal and (b) transversal directions versus applied stress during neutron diffraction  
 506 experiments.

507 One can notice the linear deviation of the hardest  $\{11.2\}_{//}$  grains (Fig. 12) with a change in  
 508 relative activity between prismatic  $P\langle a \rangle$  and basal  $B\langle a \rangle$  slips (Fig. 11). Other orientations such

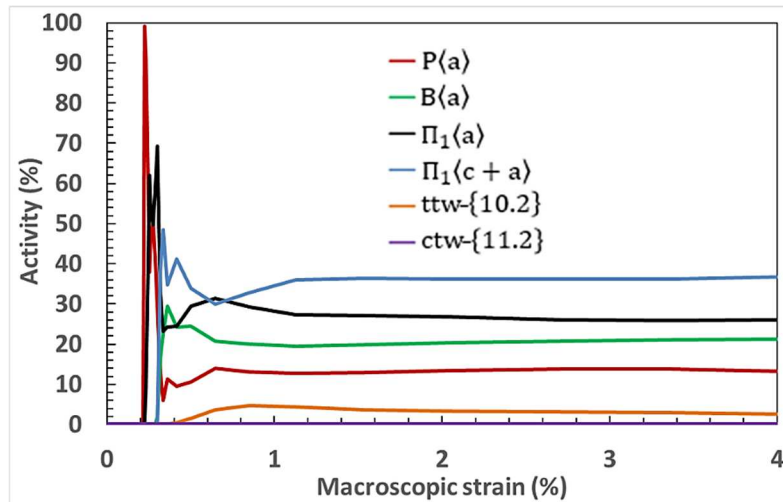
509 as  $\{10.3\}_{//}$  and  $\{00.2\}_{//}$  yield only at 355 MPa with the activation of the pyramidal mode  
510  $\Pi_1\langle c + a \rangle$  or  $\{11.2\}$  twinning deformation. These  $\{hk.l\}_{//}$  grains have the  $\vec{c}$ -axis nearly  
511 aligned along the LD and they are poorly orientated for the soft  $\langle a \rangle$  slip mechanism to be  
512 activated. In the LD, the largest compressive residual strains are observed for  $\{10.1\}_{//}$  and  
513  $\{20.1\}_{//}$  grains ( $\sim -620 \mu\epsilon$  and  $\sim -399 \mu\epsilon$  at 5 % of total strain, respectively). Similar  
514 behaviors can be seen along  $\{10.0\}_{//}$ ,  $\{10.2\}_{//}$  and  $\{11.0\}_{//}$  orientations, although the lattice  
515 strains are relatively smaller:  $\sim -246 \mu\epsilon$ ,  $\sim -312 \mu\epsilon$  and  $\sim -183 \mu\epsilon$ , respectively. These  
516 behaviors are well captured by the model except for  $\{10.2\}_{//}$  orientation where a shift of 335  
517  $\mu\epsilon$  can be noticed between the experimental and simulated results. As shown in Fig. 12.a, the  
518  $\{00.2\}_{//}$  grain orientation develops significant tensile residual lattice strains ( $\sim 760 \mu\epsilon$ ) during  
519 the first 1 % of total strain. This indicates that the load is redistributed towards the plastically  
520 harder grains like the  $\{00.2\}_{//}$  ones after the initiation of plasticity. This phenomenon is  
521 expected since all these grains are poorly orientated for the soft  $\langle a \rangle$  slip mechanisms. A  
522 similar behavior is observed for the  $\{10.3\}_{//}$  grains orientation relatively close to the  $\{00.2\}_{//}$   
523 one, although the experimental lattice strains are relatively smaller: 570  $\mu\epsilon$ . This set of grains  
524 is also unfavorable to  $\langle a \rangle$  slips. Their residual strain evolution is well described by the  
525 numerical simulations.

526 In the TD (Fig. 12.b), the model predicts the evolution of intergranular strains in the majority  
527 of the  $\{hk.l\}$  orientations studied even if some discrepancies are observed. All  $\{hk.l\}_{\perp}$  grains  
528 present an opposite behavior to that observed in the longitudinal direction, highlighting the  
529 Poisson effect. The residual lattice strains evolution of  $\{11.0\}_{\perp}$  reflection is under-predicted.  
530 A large gap of 450  $\mu\epsilon$  is observed between the experimental and simulated results, beyond 2.3  
531 % of total strain. For  $\{20.1\}_{\perp}$  grains, the model captures the tensile state, although the state is  
532 underestimated by  $\sim 250 \mu\epsilon$  at 3 % of total strain. Nevertheless, for the majority of

533 orientations, in particular:  $\{10.0\}_\perp$ ,  $\{00.2\}_\perp$ ,  $\{10.1\}_\perp$ ,  $\{10.2\}_\perp$ ,  $\{10.3\}_\perp$  and  $\{11.2\}_\perp$ , the  
534 predictions are fairly accurate with the exception of the  $\{11.0\}_\perp$  reflection. Therefore, one can  
535 only to conclude that the difference could not be attributed to a lack of description of the  
536 hardening. In our opinion, this difference could be explained by the presence of  
537 heterogeneities in the set of grains participating to this orientation or by the reverse plasticity  
538 when the material is subjected to reverse loading. The differences between model calculations  
539 and measurements are more pronounced than in the  $\{hk.l\}_{//}$  grains. Similar observation has  
540 been made in previous studies [13,48].

#### 541 4.2 *Synchrotron diffraction results*

542 In contrast to neutron diffraction experiments, *in situ* synchrotron measurements have been  
543 performed during a continuous tensile test. Fig. 10.b shows the experimental and predicted  
544 bulk response. The simulated results describe with accuracy the elastic and plastic slopes up  
545 to 4 % macroscopic strain. The calculated Young's modulus of 107 GPa is very close to the  
546 experimental one of  $109 \pm 2$  GPa. These values are consistent with those found in the neutron  
547 diffraction analysis. This proves that the model parameters allow to describe the elastic  
548 behavior of the material, suggesting that the elastic constants used in the modeling are quite  
549 reasonable with a well-described crystallographic texture. The yield stress is 240 MPa with  
550 the prismatic slip P(a) as the main deformation mode (Fig. 13).



551

552 **Fig. 13:** Relative activities of the different active deformation modes for uniaxial tensile  
 553 loading during synchrotron diffraction experiments

554 Its initial CRSS value (110 MPa, see Table 3) is consistent with that found for N-sample and  
 555 more generally with  $\alpha$ -titanium [64–68].

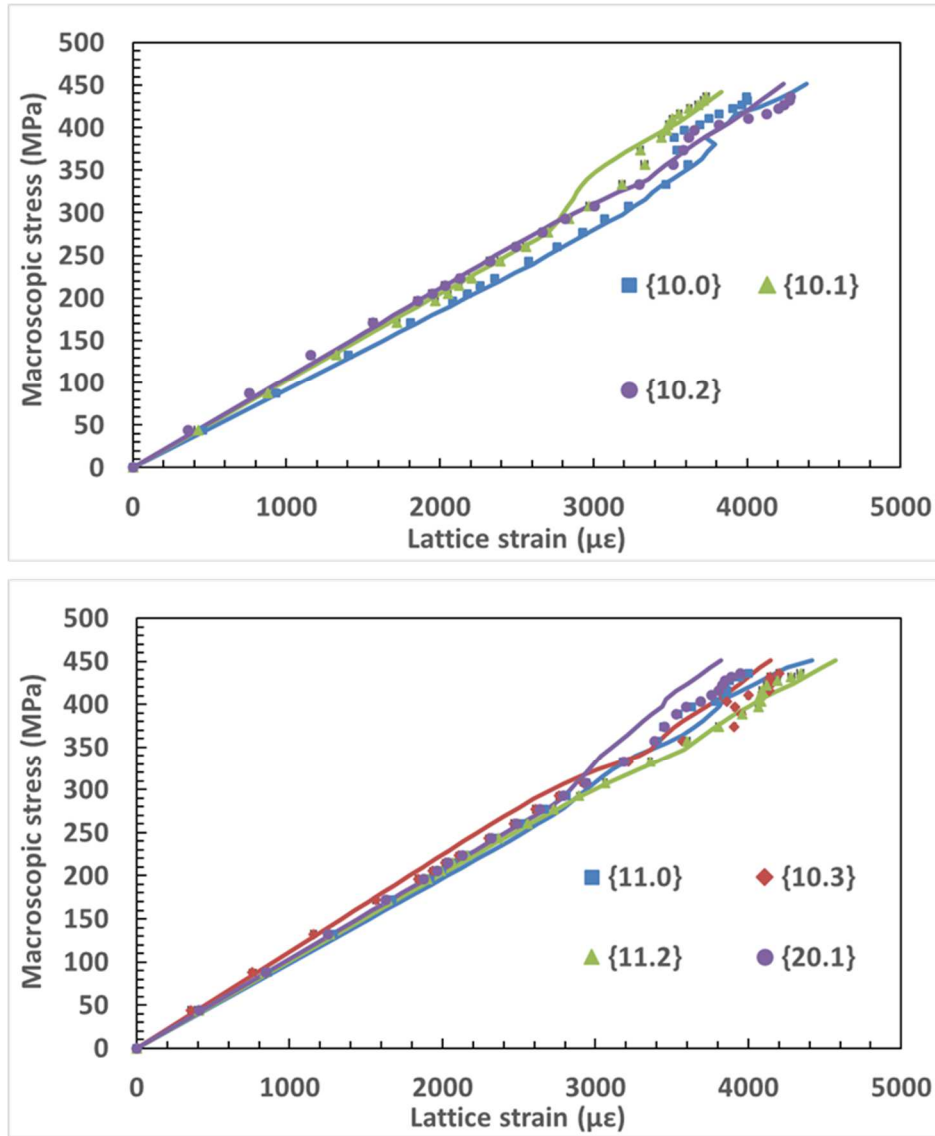
556 Concerning the prediction of lattice strain evolution, only the results obtained in the  
 557 longitudinal direction were retained. Indeed, for a given  $\{\mathbf{hk.l}\}_{//}$  reflection, all diffracting  
 558 grains have the same orientation of the  $\vec{c}$ -axis with respect to the tensile direction. They have  
 559 more-or-less the same deformation history and the Schmid factors for each deformation  
 560 system are the same for these grains. The grains which participate in the  $\{\mathbf{hk.l}\}_{\perp}$  reflection  
 561 have a wide range of crystallographic orientations with respect to the loading axis. In other  
 562 words, a rotation of a grain around an axis perpendicular to the loading direction highly  
 563 changes the stiffness in the tensile direction and, consequently, the strain and stress state in the  
 564 grain. The lattice strain of the diffracting grains, for the considered  $\{\mathbf{hk.l}\}$  reflection, shows  
 565 large variation.

566 The mechanical response in the TD is very sensitive to the texture and the elastic strain  
 567 measured is thus the average over a set of diffracting grains with different strain history.  
 568 Many authors have already pointed out this problem [11,13,70,71]. This can explain the more

569 pronounced discrepancies noticed between calculations and measurements in the direction  
570 perpendicular to the tensile axis in the [section 4. 1](#). Moreover, the number of diffracting grains  
571 in X-ray synchrotron experiments is weak as compared to neutron diffraction experiments due  
572 to the reduced gauge volume. Therefore, the lattice strains determined with synchrotron X-ray  
573 radiation are less representative of the polycrystal due to a smaller number of crystallites  
574 contributing to the diffracting volume.

575 The comparison with the homogenization approach is then more problematic especially in the  
576 TD. In the LD, this problem remains present but, for a given  $\{hk.l\}_{//}$  reflection, all the grains  
577 have an identical lattice plane normal in a direction parallel to the tensile axis with a very  
578 similar mechanical state for these grains in plasticity. The range of lattice strains for all grains  
579 in the diffracting volume along the longitudinal direction is lower than the range of the strains  
580 for the transverse direction within a given  $\{hk.l\}$  family. This compensates somehow the lack  
581 of statistics met with the gauge volume. Therefore, the focus is made on the internal strain  
582 evolution in the LD. The lattice strain evolution is reasonably predicted along LD ([Fig. 14](#)). In  
583 the elastic regime, a linear evolution is observed whose slope depends on the grain family due  
584 to the elastic anisotropy, each group of grains shows a more or less stiff behavior.





585

586 **Fig. 14:** Experimental (symbols) and predicted (lines) lattice strain evolution along LD for  
 587 tensile test during synchrotron diffraction experiments

588 For example,  $\{10.0\}_{//}$  grains family, which exhibit the most compliant behavior with the  
 589 lowest elasticity modulus  $E_{hk.l}$  (Table 5), accumulate the largest elastic lattice strains as  
 590 compared to those met for  $\{1.01\}_{//}$  and  $\{10.2\}_{//}$  reflections. The elasticity modulus  $E_{hk.l}$  are  
 591 very well predicted by the model.

592 **Table 5:** Measured and predicted elasticity modulus  $E_{hk.l}$  (GPa) for different reflections  $\{hk.l\}_{//}$

$\{10.0\}$	$\{10.1\}$	$\{10.2\}$	$\{11.0\}$
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<b>Experimental</b>	$93 \pm 0.8$	$103.2 \pm 1$	$104.2 \pm 1.8$	$97.8 \pm 1.4$
<b>Model</b>	92.2	102.5	105.8	98.6
	<b>{10.3}</b>	<b>{11.2}</b>	<b>{20.1}</b>	–
<b>Experimental</b>	$113.6 \pm 2$	$104 \pm 1$	$100 \pm 1.4$	–
<b>Model</b>	112.3	103.8	101.6	–

593

594 In general, the simulations exhibit a reasonable agreement with the experiment in terms of  
595 both trends and magnitudes. The relative activities of the deformation modes are plotted in  
596 **Fig. 13**. The  $\{10.0\}_{//}$  and  $\{11.0\}_{//}$  grain families yield first at 240 MPa. Such micro-yielding  
597 is correctly reproduced by the model, and it is clear that this first inflection is induced by the  
598 activation of prismatic slip  $P\langle a \rangle$ . At 260 MPa, the  $\{20.1\}_{//}$  and  $\{10.1\}_{//}$  grain families begins  
599 to shed load due to the activation of pyramid slip  $\Pi_1\langle a \rangle$ . A third inflection taking place at 306  
600 MPa is observed for the  $\{10.2\}_{//}$  and  $\{11.2\}_{//}$  orientations. This inflection is a result of the  
601 activity of basal slip  $B\langle a \rangle$ . A significant load partitioning takes place later in  $\{10.3\}_{//}$  grains,  
602 and is associated with the noteworthy activity of pyramidal  $\Pi_1\langle c + a \rangle$ . This slip mode  
603 becomes very quickly the major slip system governing the material plasticity until 4 % of the  
604 total strain (**Fig. 13**).

605 The effect of crystallographic texture difference between N- and S-samples is clearly visible  
606 in the evolution of relative activities of the different active deformation systems. In the N-  
607 sample, the majority of the grains have the  $\vec{c}$ -axis nearly perpendicular to the loading axis and  
608 they are favorably oriented for the prismatic slip  $P\langle a \rangle$  while in the S-sample, the rolling  
609 texture induces the significant activity of pyramidal  $\Pi_1\langle c + a \rangle$  (see **Fig. 11** and **Fig. 13**). At ~  
610 365 MPa, the lattice strain relief in the  $\{10.0\}_{//}$  grains is caused by the  $\{10.2\}$  tensile  
611 twinning inducing a stress relaxation. This observation has been made in previous works [71–

612 [73], and is well captured by the simulations. It is noted that  $\{11.2\}$  compressive twinning  
613 during the deformation is almost negligible, which is consistent with the initial  
614 crystallographic texture of the S-sample (Fig. 7).

615 For diffraction measurements, the residual strains usually show poorer agreement between  
616 measurements and simulation results than the *in situ* strains measured under a mechanical  
617 load. This can be explained by the fact that the model does not take into account slip activity  
618 [70] or twinning-detwinning [74] one during unloading (i.e the reverse plasticity, when the  
619 material is subjected to reverse loading, is not predicted by the model). The experimental  
620 macroscopic stress-strain curves show actually a slight reverse plasticity during the unloads,  
621 especially for large macroscopic strain values (see Fig. 10), for stress values close to zero.  
622 Some discrepancies between the model predictions and the measurements could therefore be  
623 expected. Despite this limitation, residual strain evolution is well described by the numerical  
624 simulations for the majority of  $\{hk.l\}$  orientations along the LD and TD (see Fig. 12). The  
625 observed discrepancies ( $\{10.2\}_{//}$ ,  $\{11.0\}_{\perp}$  for example) between the model predictions and the  
626 measurements could be partially explained by this lack of description. On the other hand, the  
627 main limitation of synchrotron is the small gauge volume resulting in a limited number of  
628 diffracting grains for a given reflection and an exacerbated sensitivity of the results to spatial  
629 heterogeneities. The comparison with a homogenization approach becomes then more  
630 problematic. In our study, in order to overcome this problem, only longitudinal strains have  
631 been analyzed and intensity of each diffraction ring has been integrated within "cake shape"  
632 sectors defined by  $\beta \pm 10^\circ$ . The arithmetic mean values of the d-spacings were subsequently  
633 calculated from the averaging of the symmetrically equivalent  $\gamma$  and  $\gamma + 180^\circ$  angles (see  
634 section 2.3). Moreover, the ratios between the CRSS of the different deformation modes with  
635 respect to the one of the  $P\langle a \rangle$  family are in agreement with the ranges identified with neutron  
636 diffraction. These results are in accordance with those obtained, for example, by Barkia et al.

637 [67] by TEM (slip trace analysis) or Amouzou et al. [68] with an EVPSC model. The same  
638 activation sequence combination has been used for these two experiments. Therefore, we can  
639 deduce that the longitudinal lattice strains determined with synchrotron X-ray radiation are  
640 representative due to a sufficient number of crystallites contributing to the diffracting volume  
641 for the LD.

642 Nevertheless, due to the crystallographic texture, the activity rates for the different  
643 deformation modes are not the same according to the samples of this study.

## 644 5. Conclusion

645 In this work, the evolution of lattice strains and texture under mechanical loading in  $\alpha$ -Ti alloy  
646 has been examined using two different techniques: neutron and X-ray synchrotron  
647 diffractions. These results have been compared with the prediction of EPSC model and the  
648 elastoplastic deformation has been studied at the different scales. The major conclusions are  
649 summarized as below:

- 650 ➤ An extended EPSC model taking into account twin domain reorientation and  
651 associated stress relaxation has been developed. On the basis of this study, the relative  
652 activity of slip and twinning deformation modes occurring in the samples have been  
653 determined and analyzed. By tuning the hardening coefficient and the initial CRSS, it  
654 is shown that the model predictions, in terms of lattice strain evolution (8 and 7  
655 different reflections for neutron and synchrotron diffraction, respectively) and  
656 macroscopic stress-strain curves are in good agreement with the experimental data.  
657 The lattice strain relief caused by the twinning is well captured by the simulations.
- 658 ➤ Our work highlights the strong crystallographic texture effect onto the deformations  
659 mechanisms activation sequence of N- and S-samples. In the N-sample, the majority  
660 of the grains have the  $\vec{c}$ -axis nearly perpendicular to the loading axis and they are

661 favorably oriented for the prismatic slip  $P\langle a \rangle$  (and for the tensile twinning  
662  $\{10.2\}\langle 10.\bar{1} \rangle$ ) while in the S-sample, the rolling texture induces the significant  
663 activity of pyramidal  $\Pi_1\langle c + a \rangle$ .

664 ➤ X-ray synchrotron source offers fast rates of data acquisition (few seconds for X-rays  
665 and about 40 minutes for neutron diffraction in this work) enabling real time and *in*  
666 *situ* strain distribution measurements during continuous mechanical loading. Despite a  
667 small irradiated volume for X-rays, the inflections of  $\{hk.l\}$  orientations due to the slip  
668 or twinning deformation modes are relatively well reproduced by the model. This  
669 agreement is only obtained in the LD with intensity of each diffraction ring has been  
670 integrated within "cake shape" sectors defined by  $\beta \pm 10^\circ$ . The arithmetic mean values  
671 of the d-spacings were calculated from the averaging of the symmetrically equivalent  $\gamma$   
672 and  $\gamma + 180^\circ$  angles.

673 ➤ The advantage of neutron diffraction techniques is notably related to a substantial and  
674 representative scattering volume of  $4 \times 4 \times 4 \text{ mm}^3$ . Despite the fact that the model  
675 does not take into account slip activity or twinning-detwinning one during unloading,  
676 residual strain evolution is well described by the numerical simulations for the  
677 majority of  $\{hk.l\}$  orientations along LD and TD.

678 ➤ The comparison between the extended EPSC model with *in situ* or *post mortem*  
679 characterization give the same trends: the  $\langle a \rangle$  slip on prismatic planes is the easiest  
680 deformation mechanism. Pyramidal  $\langle a \rangle$  slip is the second easiest and basal  $\langle a \rangle$  slip the  
681 third.  $\langle c + a \rangle$  slip is regarded as more difficult than  $\langle a \rangle$  slip mechanisms which result  
682 in twinning to accommodate strain along the  $\vec{c}$ - axis.

683

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