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1 **Influence of plastic anisotropy and strain path on strain-induced phase transformation**
2 **of cobalt**

3

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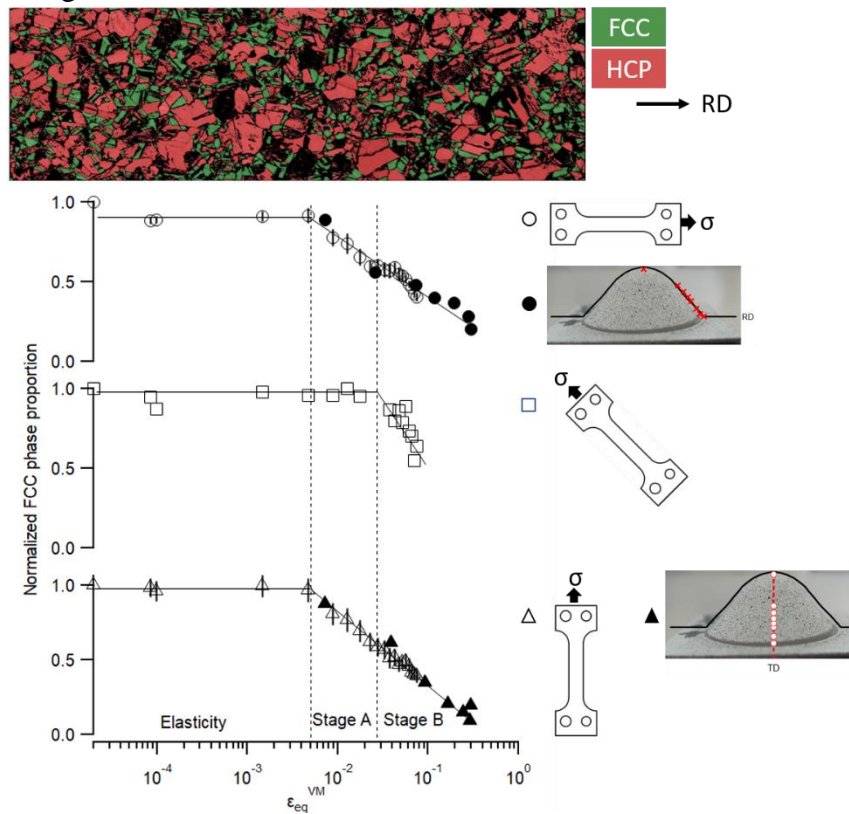
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9

10 On behalf of all authors, the corresponding author states that there is no conflict of interest.

11

12 **Graphical abstract:** Evolution of metastable cobalt FCC volume proportion during
13 mechanical loading.



14

15 **Abstract**

16 The objective of this paper is to contribute to a better understanding of the deformation
17 mechanisms of a rolled polycrystalline cobalt sheet. In order to achieve this goal, a particular

18 attention is given to the strain-induced phase transformation and the forming parameters on
19 which it may depend such as the plastic anisotropy or the loading path.

20 *In situ* phase quantification by X-ray diffraction during tensile tests, on samples cut out from a
21 cold rolled sheet material in three different directions (at 0°, 45° and 90° from the rolling
22 direction), has enabled to study the influence of plastic anisotropy on the strain-induced phase
23 transformation of metastable retained Face Centered Cubic phase to Hexagonal Close Packed
24 phase. Results show that the martensitic phase transformation can be activated by basal slip in
25 the first stage of plasticity but it also indicates that the latter is delayed when sample is
26 strained in tension for an angle of 45° to the rolling direction. *Post mortem* analyses on a
27 stamped sample by the Nakazima test have revealed that the phase transformation continues
28 for higher strain levels. A particular interest is paid to the coupling between strain-hardening
29 mechanisms and phase transformation and is discussed given the plastic anisotropy of cobalt
30 aggregate.

31

32 **Keywords:** cobalt, mechanical properties, anisotropy, phase transformation, X-ray diffraction

33

34 **1. Introduction**

35 Work-hardening mechanisms of Hexagonal Closed-Packed (HCP) cobalt are quite specific
36 due to the c/a ratio close to the ideal value, the high melting temperature [1] and the low
37 stacking fault energy [2]. Basal dislocation glide on the $\{00.1\}\langle\bar{1}2.0\rangle$ crystallographic
38 system is the first stage of cobalt work hardening and it has been extensively studied in the
39 50s and 60s by Seeger *et al.*[3–5]. Due to the limited number of available slip systems, the
40 plastic deformation is quickly accommodated by the twinning process as the second stage of

41 work hardening. More recently, in the 2010s, Martinez *et al.* [6–9] and Zhu *et al.* [10,11] have
42 studied more specifically the key-role of twinning to accommodate plastic deformation.

43 HCP cobalt presents an allotropic transformation to Face Centered-Cubic (FCC) for
44 temperatures higher than 440 °C while the reverse martensitic transformation temperature is
45 around 330 °C, exhibiting a hysteresis. This transformation is not fully reversible and the
46 metastable FCC phase remains generally present at room temperature. This allotropic
47 transformation, caused by temperature, has been extensively studied in the 1970s and from the
48 1990s to 2010 [12–31]. The retained FCC phase is mechanically unstable under stress unless
49 it has undergone a specific heat treatment under controlled time and temperature conditions
50 (long time combined with high temperatures) [8,32]. The properties of cobalt, both
51 mechanical and magnetic, have also been extensively studied [1,32–39]. Some original effects
52 such as sensitivity to size effects [8,39,40] or non-linear elasticity [38,41] have been the
53 subject of recent studies. However, only few studies have focused on two-phase effect with
54 the exception of one which showed that the presence of the residual FCC phase was beneficial
55 to the mechanical properties [33]. This brief study does not mention the mechanical stability
56 of the microstructure with heat treatments and was therefore not interested in the possible
57 phase transformation induced by the mechanical deformation.

58 The strain-induced FCC to HCP phase transformation in cobalt has rarely been investigated
59 since Sanderson's work in the 1970s [32]. Only a few recent works on thin films [42] and on
60 the influence of temperature [43], deal nevertheless with this mechanism [32,42,43].
61 Concerning the cobalt alloys, the study on a Co-Ni-Cr-Mo alloy shows a correlation between
62 deformation modes, stacking fault energy and strain-induced phase transformation [44]. On
63 Co-Cu alloys, a full FCC to HCP transformation has been observed for a severe plastic
64 deformation process [45]. These investigations agree that the residual FCC phase can be

65 transformed into HCP phase by plastic deformation at room temperature, provided that it has
66 not been stabilized by a heat treatment.

67 Dubos *et al.* [46,47] have investigated the size effects in complex forming processes of
68 different high-purity metals. For mechanically stabilized microstructure of polycrystalline
69 cobalt, first results exhibit a strain path dependence of size effects during stamping tests. This
70 deformation process can actually be used for the microforming of miniaturized systems in
71 which cobalt can be a good candidate due to its specific mechanical and magnetic properties.
72 The fields of application can be varied such as sensors, electronics, micro-mechanics,
73 medical, *etc.*

74 These previous works clearly show a renewed interest in this material, despite the complexity
75 of its deformation mechanisms and the little-studied phenomena such as plasticity-induced
76 phase transformation when the microstructure is not mastered beforehand.

77

78 Plastic anisotropy is an extremely important factor to be considered in sheet metal forming.
79 The most studied HCP phase metals (i.e. magnesium, zirconium and titanium) display strong
80 plastic anisotropy which results in a Lankford coefficient varying between 2 and 4 [48]. This
81 plastic anisotropy directly results from the marked rolling texture of HCP metal sheets [49].
82 For polycrystalline cobalt, Lankford coefficient is around 0.92 (weak plastic anisotropy).
83 Nevertheless, it is strongly influenced by the microstructure, notably the grain size [47].

84 The main aim of this experimental study is to contribute to the knowledge and the
85 understanding of the strain-induced phase transformation in cobalt when the FCC retained
86 phase is mechanically unstable. After a detailed description of the experimental methods,
87 focusing on X-Ray Diffraction (XRD) phase quantification and mechanical tests carried out

88 (tensile and stamping tests), results are given to highlight the influence of plastic anisotropy
89 and the strain path deviation on the FCC to HCP phase transformation induced by plasticity.
90 *Post mortem* phase quantifications will be presented on a stamped sample which displays a
91 high strain gradient and will be compared to those obtained by tensile tests in different
92 directions by considering von Mises equivalent strain. Finally, the plastic anisotropy effect
93 will be discussed in view of activation and transformation rate. In a second step, the influence
94 of plastic anisotropy on the main work-hardening mechanisms and on the competition
95 between twinning and phase transformation will be raised.

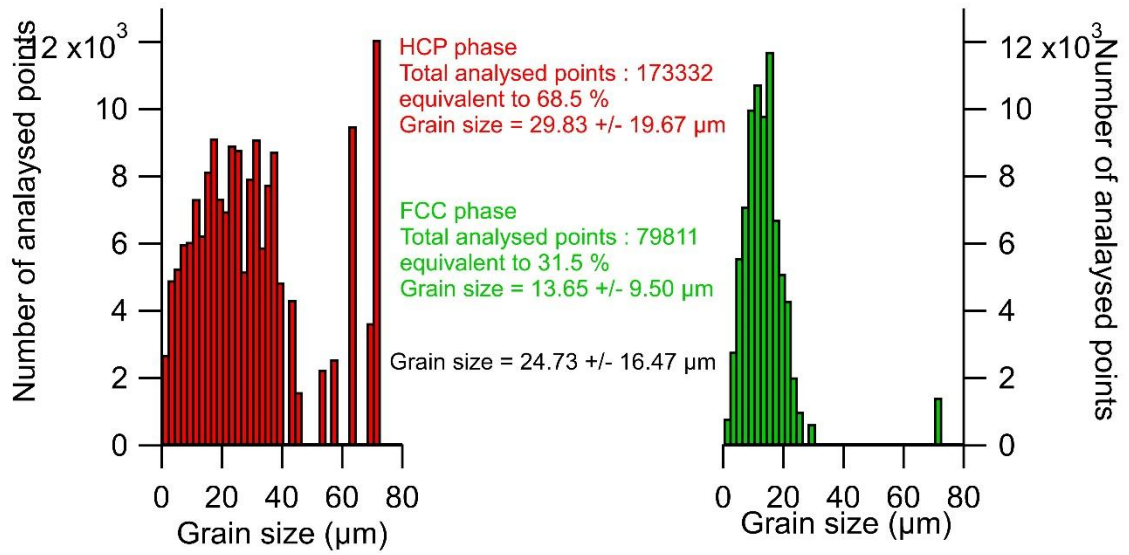
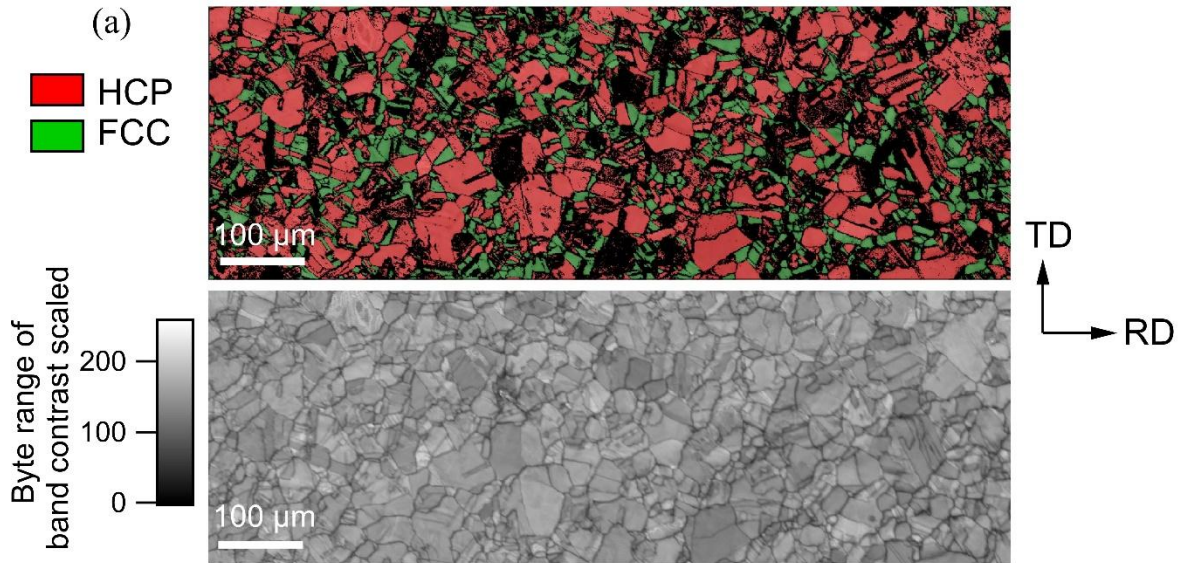
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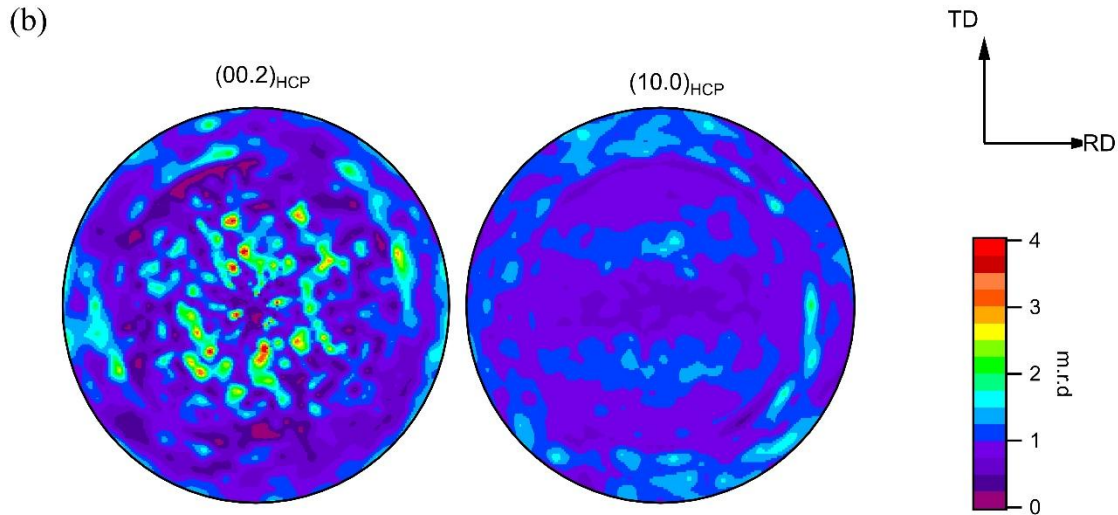
97 **2. Material and methods**

98 Commercial polycrystalline cobalt sheets (Goodfellow Company) of high-purity (99.9 wt. %),
99 cold-rolled to 0.5 mm in thickness in a recrystallized state, were used for the present study.
100 Electron Back Scattered Diffraction (EBSD) mapping has been performed (**Fig. 1 (a)**) in
101 order to obtain initial grain size and phase cartographies. The initial ratio between the two
102 phases is around 70 % HCP / 30 % FCC with corresponding grains sizes of $30 \pm 20 \mu\text{m}$ and
103 $14 \pm 10 \mu\text{m}$.

104 The calculation of the grain size is performed on each phase, only with the indexed pixels.
105 There seems to be no correspondence between the band contrast and the non-indexed pixels.
106 This confirms the recrystallized state of the sheet after cold rolling and the absence of
107 significant residual stresses.

108





110

111 **Figure 1:** Microstructural analysis of the as-received material: (a) EBSD map of phase
 112 distribution and band contrast. Distribution of grain size for each phase. HCP phase is colored
 113 in red and FCC phase in green. (b) XRD crystallographic textures of the HCP phase
 114 determined from the orientation distribution functions calculated by the WIMW method
 115 implanted in the BEARTEX software.

116

117 Crystallographic textures and retained FCC phase ratios, at room temperature, were
 118 determined by XRD technique. A four-circle Seifert XRD3003 PTS diffractometer equipped
 119 with a Euler cradle, a Position Sensitive Detector (PSD) with a 2θ effective windows of 8.6° ,
 120 a 1 mm diameter collimator and an IDS camera (for sample positioning in the center of the
 121 goniometer), was used. A Cr anode ($\text{CrK}_{\alpha 1+2}$ radiation), operating at 30 kV and 35 mA, was
 122 used (wavelength $\lambda = 2.28964 \text{ \AA}$). The K_β component of the Cr radiation was eliminated using
 123 a vanadium filter installed in front of the detector.

124 Experimental XRD peak profiles were fitted by a Pseudo-Voigt function which takes into
 125 account the $\text{K}_{\alpha 1}$ - $\text{K}_{\alpha 2}$ doublet [50]. For each diffraction peak, background was modeled through
 126 a 2^{nd} order polynomial function. Prior to peak profile analysis, angular corrections (Lorentz,
 127 polarization and absorption factors) have also been applied [51].

128 **Fig. 1 (b)** shows recalculated $\{00.2\}_{\text{HCP}}$ and $\{10.0\}_{\text{HCP}}$ Pole Figures (PFs) of the as-received
 129 sample. The crystallographic textures have been determined from the Orientation Distribution
 130 Functions (ODF) calculated by the WIMV method implemented in the BEARTEX software
 131 [52], and were obtained from $\{10.0\}_{\text{HCP}}$, $\{00.2\}_{\text{HCP}}$, $\{10.1\}_{\text{HCP}}$ and $\{10.2\}_{\text{HCP}}$ experimental
 132 PFs with an average error coefficient of about 0.08. This initial state displays a slight centered
 133 basal reinforcement of ($\{00.2\}_{\text{HCP}}$ on Fig. 1 (b)) with symmetrical intensity distribution of
 134 ($\{00.2\}_{\text{HCP}}$ and $\{10.0\}_{\text{HCP}}$ on Fig. 1 (b)) representative of a rolling texture for hexagonal
 135 materials with a c/a ratio close to 1.633. The central pole of $\{00.2\}_{\text{HCP}}$ in Fig. 1 (b) is
 136 distributed up to an angle of 43.1° characteristic of the habit plane between the two phases
 137 considering the Shoji-Nishiyama's relation [10].

138

139 The FCC phase volume fraction (denoted f^{FCC}) has been determined for each measurement
 140 point using the equation (1) as formulated by Shang *et al.* [53]:

$$f^{\text{FCC}} = \left(1 + \frac{I_{\text{hkl}}^{\text{HCP}} R_{\text{hkl}}^{\text{FCC}}}{I_{\text{hkl}}^{\text{FCC}} R_{\text{hkl}}^{\text{HCP}}} \right)^{-1} \quad (1)$$

141 where I_{hkl} is the integrated intensity of the corresponding $\{hkl\}$ peak. R_{hkl} is the reflectivity of
 142 the lattice plane $\{hkl\}$, and is given by the following equation (2) introduced in the literature
 143 [54]:

$$R_{\text{hkl}} = \frac{1}{v^2} |F_{\text{hkl}}|^2 \left(\frac{1 + \cos^2(2\theta)}{\sin^2(2\theta)\cos(\theta)} \right) m \frac{e^{-2M}}{\mu} \quad (2)$$

144 where v is the volume of the crystal unit cell (in \AA^3), F_{hkl} is the structure factor, 2θ is the
 145 considered Bragg angle (in rad), m is the $\{hkl\}$ planes multiplicity factor, μ is the linear
 146 absorption coefficient (in cm^{-1}) and e^{-2M} is the Debye-Waller factor [1,55].

147 The volume fraction of the phases was calculated from several groups of family planes,
148 combining $\{10.0\}_{\text{HCP}}$, $\{00.2\}_{\text{HCP}}$, $\{10.1\}_{\text{HCP}}$, $\{10.2\}_{\text{HCP}}$ and $\{111\}_{\text{FCC}}$, $\{200\}_{\text{FCC}}$, and
149 averaged.

150 *In situ* monotonic uniaxial tensile tests were carried out, at room temperature, to study the
151 influence of elastoplastic strain on the allotropic transformation. These mechanical tests were
152 performed on a DEBEN device with a load cell of 5 kN capacity and controlled by the
153 displacement between clamping jaws. The dog bone shaped tensile specimens were machined
154 by electrical discharge machining and designed a gauge section of 26 mm in length, 7 mm in
155 width and 0.5 mm in thickness in three different directions having an angle α with respect to
156 the Rolling Direction (RD) of the sheet: $0^\circ/\text{RD}$, $45^\circ/\text{RD}$ and $90^\circ/\text{RD}$, latter corresponding thus
157 to the Transverse Direction (TD).

158 In order to quantify the evolution of the phase ratio during the tensile loading, the samples
159 were strained stepwise up to 7.8 % with a strain rate of 0.2 mm/min (quasi-static deformation
160 hypothesis). At each straining step, and after a stress relaxation of about 20 minutes ensuring
161 stable mechanical fields over the entire diffracting volume, X-ray diffraction analyses were
162 carried out at each measurement point.

163 To obtain the macroscopic mechanical response, a second mechanical test series was achieved
164 where the specimens were continuously strained up to 7.8 % with a strain rate of 0.2 mm/min.
165 On one side of the sample, longitudinal and transverse deformations were measured with
166 strain gauges in order to obtain the Young modulus E and the Poisson's coefficient ν . On the
167 other side of the specimen, a speckle was deposited and images were recorded during the test.
168 Digital Image Correlation (DIC) has then been processed to obtain the macroscopic total
169 strain fields during the mechanical tests. Images were recorded with a Point-Grey GS3-U3
170 camera with 2448×2048 px² resolution and a lens of 35 mm. DIC has been performed with

171 VIC 2D software using a region of interest of 155×37 px² (i.e. 26.35×6.29 mm² with a pixel
 172 size of 0.17 mm), a subset of 9×7 px² and a grid step of 1×1 px². Planar strain fields were
 173 computed in order to obtain the ϵ_{xx} and ϵ_{yy} strain components along the tensile and the
 174 transverse directions, respectively. For each sample, the plastic anisotropy coefficient (also
 175 called Lankford) was calculated from the monotonic plastic regime with the following Eq. (3),
 176 considering a plastic incompressibility [56,57]:

$$177 \quad r_{\alpha} = \frac{\epsilon_{yy}^p}{\epsilon_{zz}^p} = - \frac{\epsilon_{yy}^p}{\epsilon_{xx}^p + \epsilon_{yy}^p} \quad (3)$$

178 α being the angle between the RD and the specimen axes.

179 With these measurements, corresponding strain paths can be characterized with the parameter
 180 β which can be expressed by:

$$181 \quad \beta_{\alpha} = \frac{\epsilon_{yy}}{\epsilon_{xx}} \quad (4)$$

182 Combining equations (3) and (4) the strain path can be directly introduced as a function of the
 183 Lankford coefficient in the following relationship:

$$184 \quad \beta_{\alpha} = - \frac{r_{\alpha}}{1+r_{\alpha}} \quad (5)$$

185 The mean Lankford coefficient r_{mean} , relative to the mean anisotropy of the sheet, and Δr ,
 186 representative of the planar anisotropy, are respectively calculated thanks to equations (6) and
 187 (7):

$$188 \quad r_{mean} = \frac{r_{0^{\circ}} + 2r_{45^{\circ}} + r_{90^{\circ}}}{4} \quad (6)$$

$$189 \quad \Delta r = \frac{r_{0^{\circ}} - 2r_{45^{\circ}} + r_{90^{\circ}}}{2} \quad (7)$$

190 Table 1 gives these coefficients for the three analyzed directions. In accordance with previous
 191 results [47], the as-received cobalt displays a mean Lankford coefficient of 0.9 indicating a
 192 mild plastic anisotropy. In this table, $|\beta - \beta_{iso}|$ represents the relative deviation of the strain path
 193 as compared to the isotropic case ($\beta_{iso} = -0.5$). The analysis of these parameters reveals a
 194 qualitative difference of the material behavior when the sample is strained at 45°/RD.

195

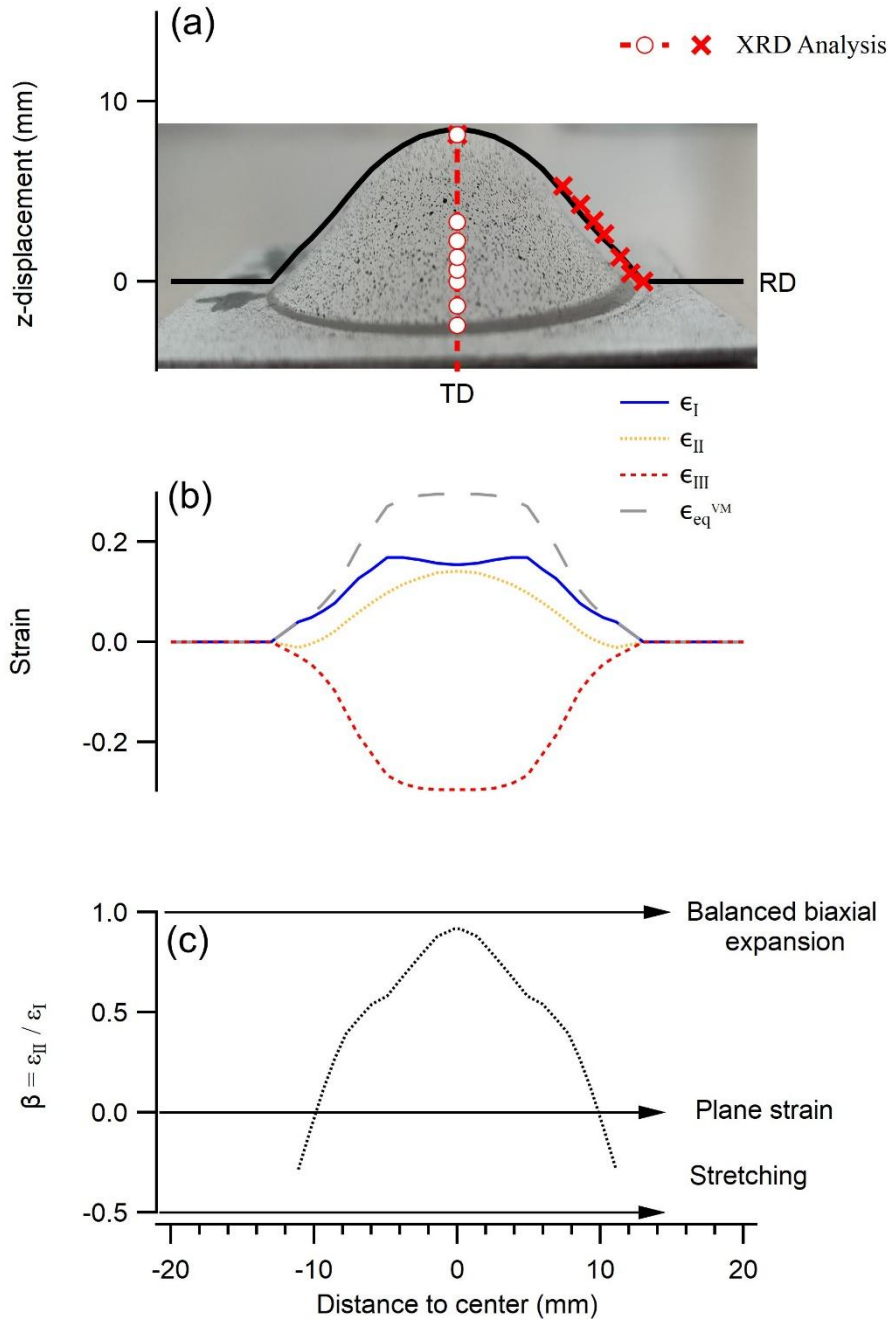
196 **Table 1:** Review of the main coefficients characterizing elastic and plastic anisotropies
 197 determined for polycrystalline specimens (E : Young modulus, ν : Poisson coefficient,
 198 α : direction of specimen collection, r : Lankford coefficient, β : strain path, $|\beta - \beta_{iso}|$: strain path
 199 deviation, r_{mean} : mean plastic anisotropy coefficient, Δr : planar plastic anisotropy coefficient).

	0°/RD (RD)	45°/RD	90°/RD (TD)
E [GPa]	167	181.3	152
$u(E)$ [GPa]	14	3.8	19
ν	0.32632	0.38573	0.38712
$u(\nu)$	0.00023	0.00069	0.00078
r_α	0.8110	0.77344	1.242
$u(r_\alpha)$	0.0034	0.00057	0.022
β	-0.4491	-0.43617	-0.5579
$u(\beta)$	0.0011	0.00018	0.0090
$ \beta - \beta_{iso} $	0.0509	0.06383	0.0579
r_{mean}	0.9000		
Δr	0.2531		

200

201 Stamping test, also named Nakazima test, is a complex biaxial loading experiment that
 202 enables to obtain various loading conditions in only one test. Nakazima tests were performed
 203 with a universal sheet metal testing machine (Zwick Roell BUP 600) on the as-received cobalt

204 sheet using a 20 mm hemispherical punch at a punch speed of 1 mm/s. The ratio of punch
205 radius on sheet thickness is large enough to assume plane-stress deformations. Blank holder
206 force was set to 3000 N to ensure no sliding. To reduce friction effect between the punch and
207 the sample, a grease/polytetrafluoroethylene/grease sandwich was employed. Strain fields
208 were measured during the tests using 3D DIC. The experimental procedures for these tests are
209 described with more details in previous works [46,47]. XRD analyses have been performed on
210 stamped samples along RD and TD in order to follow the retained FCC phase evolution under
211 a complex loading. For each measurement point, the irradiated area has been reduced (a circle
212 with a diameter of 1 mm) with a Pb masking to avoid the geometric errors associated to the
213 curved surface. **Fig. 2** shows the location of the XRD analysis points on the stamped dome
214 (a). The three principal components of the strain fields are presented along a section of the
215 stamped dome (b). ε_{III} is calculated with the plastic incompressibility assumption ($\varepsilon_{III} = -\varepsilon_I - \varepsilon_{II}$).
216 The weak plastic anisotropy, combined with residual friction, induces a slight deviation of the
217 strain path. A strain path gradient along the section of the stamped dome is revealed and must
218 be considered (c). The equibiaxiality of the loading is increased between the blank holder and
219 the top of the punch. This test allows to obtain a macroscopic equivalent strain gradient along
220 a section that could induce a composition gradient in terms of phase proportion. The
221 equivalent strain level is higher, (around 0.3), than the maximum strain that could be reached
222 with a tensile test.



223

224 **Figure 2:** Description of the Nakazima test: (a) height of the part after stamping and location
 225 of the analysis points by XRD (cross and circle marker for measurements in RD and TD,
 226 respectively), (b) decomposition of the principal components of the macroscopic strain fields
 227 (ϵ_I : in-plane major strain (along RD), ϵ_{II} : in-plane minor strain (along TD), $\epsilon_{III} = -\epsilon_I - \epsilon_{II}$: in the
 228 thickness strain and corresponding ϵ_{eq} von Mises equivalent strain), (c) evolution of the strain
 229 path β versus the location on the section.

230

231 **3. Results and discussion**

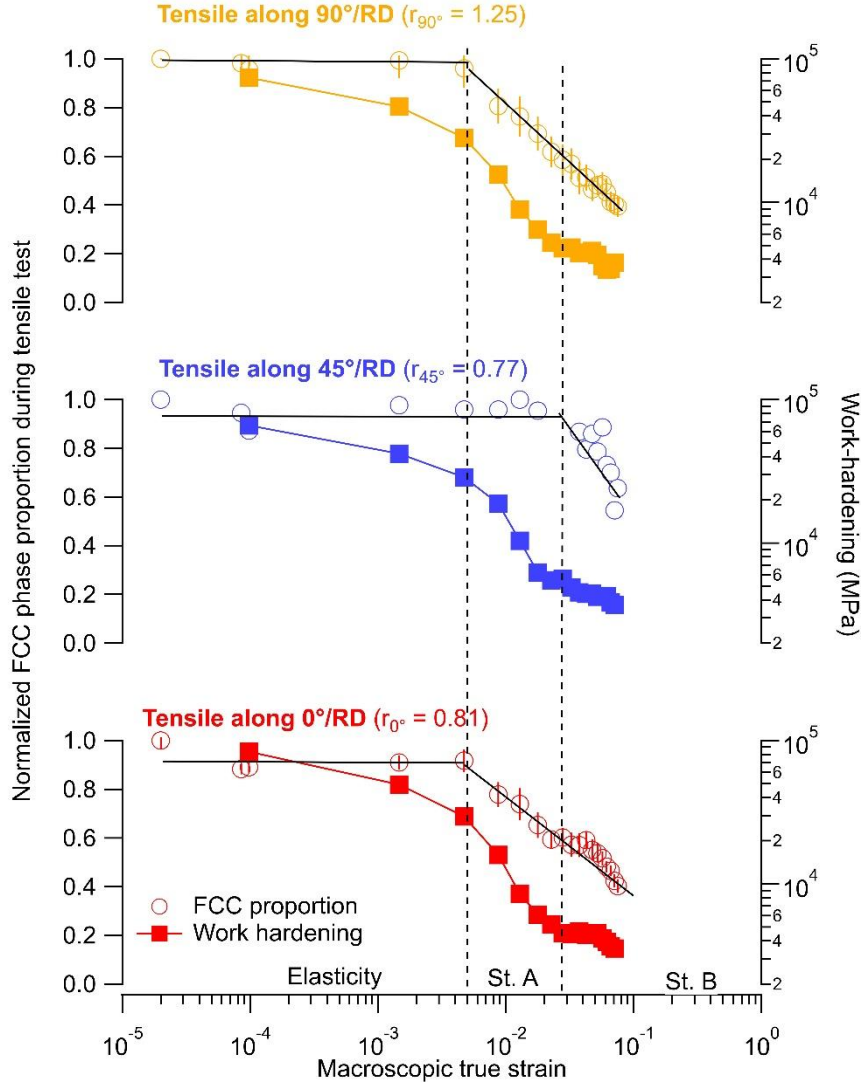
232 3.1. Influence of the plastic anisotropy

233 Retained FCC phase proportion and work-hardening rate ($\theta = d\sigma/d\varepsilon$) evolutions with the
234 macroscopic strain, ε , applied during tensile tests for the three considered directions (0° /RD,
235 45° /RD and 90° /RD) are plotted in **Fig. 3**. The calculated value of r_α is indicated on this
236 figure.

237 Work-hardening curves exhibit two distinct stages after elasticity. Basal dislocation slip is the
238 dominant mechanism in the stage A [8] while twinning mechanisms occur in stage B to
239 accommodate plastic deformation [7]. Results from **Fig. 3** show that anisotropy does not seem
240 to affect the work-hardening stages. No matter the misorientation set between the loading axis
241 and the RD, elastic strain does not allow for the phase transformation to be initiated. For $\alpha =$
242 0° and 90° , FCC to HCP phase transformation is initiated in stage A of plasticity. Beyond
243 that, the initial retained FCC phase transforms into HCP phase in a linear way to the logarithm
244 of the strain. At a macroscopic strain of 7.8 %, approximately 60 % of the initial retained FCC
245 phase is strain-induced transformed. For $\alpha = 45^\circ$, the initiation of the phase transformation is
246 delayed to the stage B. For this particular direction, twinning mechanisms seem to initiate the
247 FCC to HCP phase transformation. The plastic deformation transforms about 40 % of the
248 initially present FCC phase at 7.8 %.

249

250



251

252 **Figure 3:** FCC phase proportion (normalized to the initial retained FCC phase) and work-
 253 hardening rate evolutions under tensile testing for different angles between the load axis and
 254 the rolling direction (RD): 0° /RD (RD), 45° /RD and 90° /RD (TD°).

255

256 The evolution of the FCC phase ratio with the macroscopic strain can be summarized by the
 257 following equation:

$$258 \frac{\%FCC}{\%FCC_{init}} = \begin{cases} cst \approx 1 & \text{if } \varepsilon < \varepsilon_{init}(\alpha) \\ A(\alpha) + m(\alpha) \times \log(\varepsilon) & \text{if } \varepsilon > \varepsilon_{init}(\alpha) \end{cases} \quad (8)$$

259 where $A(\alpha)$ is a constant which depends on the orientation of the sample regarding the RD and

260 $m(\alpha)$ is the slope of the normalized phase ratio which can be assimilated to a rate of

261 transformation with the strain. $\varepsilon_{init}(\alpha)$ matches the strain at which the phase transformation
 262 initiates. These values are presented in **Table 2** for each direction.

263 **Table 2:** Coefficients of equation (8) with standard deviations (u) and linear regression
 264 coefficient (R^2) as a function of the loading axis misorientation regarding the RD.

	$\alpha = 0^\circ/\text{RD}^\circ$ (RD)	$\alpha = 45^\circ/\text{RD}$	$\alpha = 90^\circ/\text{RD}^\circ$ (TD)
m	-0.373	-0.80	-0.459
$u(m)$	0.022	0.11	0.012
A	0.027	-0.25	-0.120
$u(A)$	0.033	0.14	0.019
R^2	0.95	0.93	0.99
ε_{init}	0.00247	0.0281	0.00365

265

266 In a recent work [43], it has been shown that, for a particular direction (in this case, RD), ε_{init}
 267 increases with temperature. However, the transformation rate is independent for temperatures
 268 lower than the initiation temperature of austenitic transformation. Sanderson [32] has shown
 269 that $m(\alpha)$ is strongly affected by the initial microstructure (grain size and initial FCC
 270 proportion). Marx *et al.* [42] have highlighted that the integrated area of the $\{002\}_{\text{FCC}}$
 271 reflection decreases with macroscopic strain until a plateau is forming for strains higher than
 272 8 %. These observations tend to highlight a threshold deformation at which the residual FCC
 273 phase no longer transforms. Unfortunately, the critical macroscopic strain reached in our work
 274 for tensile tests does not allow us to confirm this phenomenon. In order to track the phase
 275 transformation for macroscopic equivalent strain higher than 7.8 %, a stamping test, whose
 276 methodology was described in the previous section, was performed.

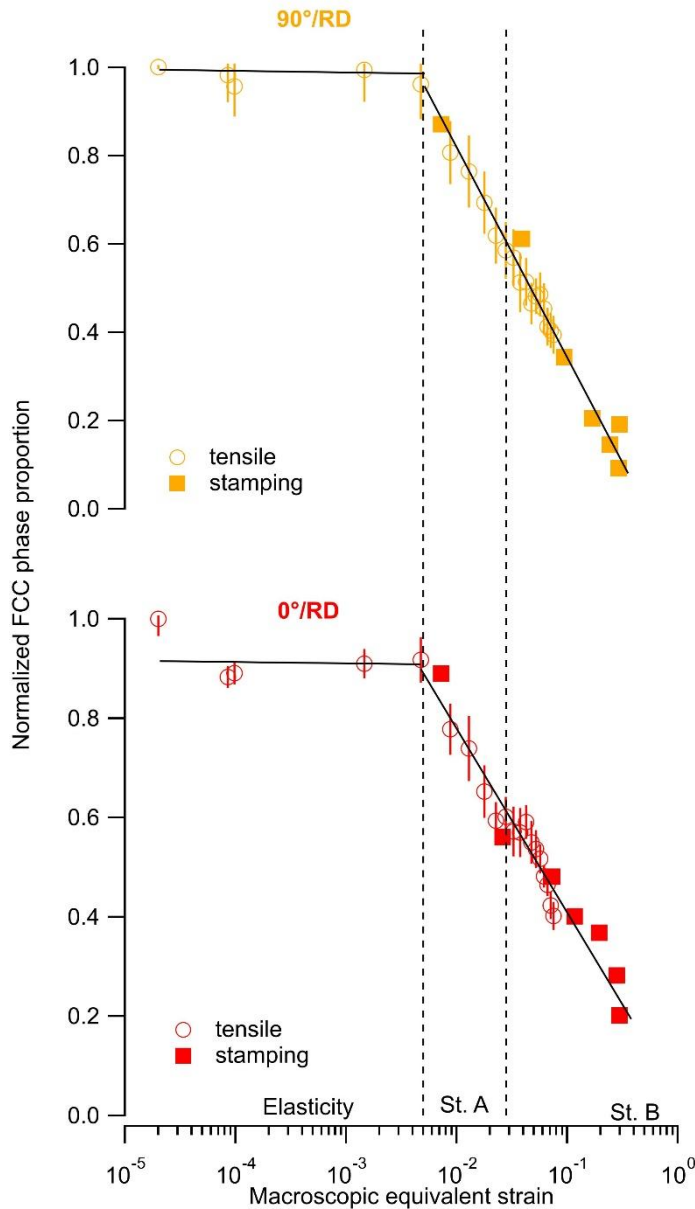
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278 3.2. Influence of the mechanical loading on the allotropic transformation

279 As shown in **Fig. 2**, *post-mortem* XRD analyses have been performed at different locations on
280 the stamped dome between the blank-holder and the top of the punch. Strain path, and
281 therefore equivalent strain level, is dependent on the chosen measurement point. In order to
282 consider the plastic anisotropy effect, phase quantification has been achieved along two
283 directions (RD and TD).

284 **Fig. 4** displays the evolution of the FCC phase ratio normalized by the initial retained FCC
285 phase versus the equivalent macroscopic von Mises strain for the two directions RD and TD
286 on the stamped sample (filled points). These results are superimposed on those obtained in
287 tension for specimens taken with an angle $\alpha = 0^\circ$ and 90° (empty points).

288



289

290 **Figure 4:** Influence of different biaxial loading scenarios on the phase transformation.
 291 Evolution of the FCC phase ratio along the rolling and transverse directions (filled squares) of
 292 a stamped sample, superposed on the *in situ* tensile test results (circles).
 293

294 The transformation rate is shown to be independent from the strain path but slightly direction-
 295 dependent. During stages A and B of plasticity, the FCC phase proportion decreases linearly
 296 with the logarithm of the von Mises' equivalent strain. The slopes are the same as those
 297 obtained with tensile tests and therefore always dependent on α angle. The maximum strain
 298 reaches about 30 % in the center of the Nakazima specimen, resulting in a transformation of

299 around 70 % of the FCC phase initially retained. Straumal *et al.* showed that a complete
300 transformation was possible in Co-Cu alloys deformed by severe plastic deformation
301 processes such as high-pressure torsion (HPT). These authors estimated that a severe plastic
302 shear strain higher than 35 % could achieve a full FCC to HCP phase transformation [45].
303 Extrapolating the evolution law obtained from our results with the stamping tests, a complete
304 transformation of the initially present FCC phase can only be expected for very high levels of
305 plastic strain, in the order of 100 %. Finally, the main point is that the strain-induced FCC to
306 HCP phase transformation continues for higher strain level than the one reached by previous
307 tensile tests.

308

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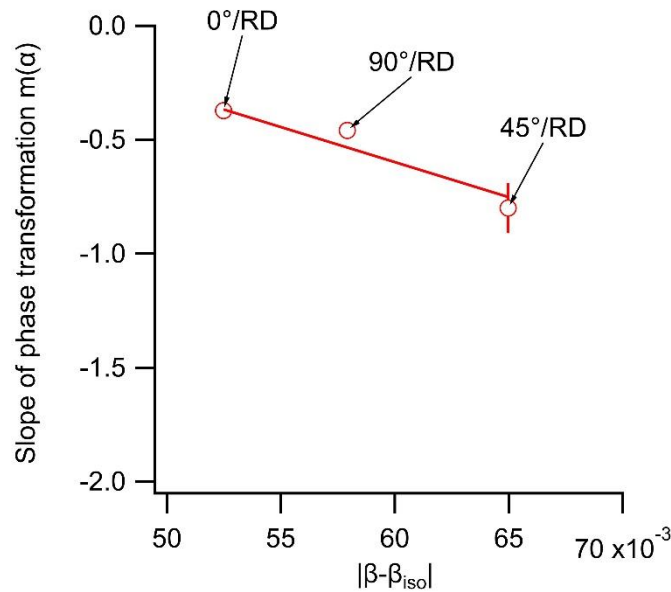
310 3.3. Relation between plastic anisotropy, strain path and phase transformation rate.

311 **Fig. 5** displays the slope of the strain-induced transformation, $m(\alpha)$, for tensile tests, plotted
312 for the three considered values of α , versus the absolute value of $\beta - \beta_{iso}$. Latter is representative
313 of the strain path deviation due to anisotropy as compared to the strain path of the isotopic
314 case. The deviation of r_α from 1 significantly affects the strain path: for a tensile loading, if r_α
315 is higher than 1, the loading conditions is close to plane strain ones, while if r_α is lower than 1,
316 the loading conditions tend to shear ones. The strain path seems to be less affected in the case
317 of a positive r_α variation (values above the isotropic case i.e. $r_\alpha = 1$) than a negative one (r_α
318 values below 1). For a value of $r_\alpha = 1.25$, the positive variation of 0.25 results in a 11
319 %deviation of the strain path. For a value of $r_\alpha = 0.75$, the deviation is negative and equal to -
320 0.25 and results in a 14 %deviation of the strain path. These two examples are respectively
321 representative of the cases with $\alpha = 90^\circ$ and $\alpha = 45^\circ$.

322 $m(\alpha)$ exhibits a linear decrease with increasing strain path deviation. **Fig. 5** illustrates the
 323 strong impact of $|\beta-\beta_{iso}|$ value on $m(\alpha)$. For $\alpha = 45^\circ$, which presents a slightly lower negative
 324 value of r_α variation than for $\alpha = 0^\circ$ and a sign variation from the isotropic case opposite to
 325 r_{90° , the transformation rate is almost twice higher. This tends to be in line with the results
 326 obtained by Straumal *et al.* [45]. Indeed, for shear loading conditions, the deformation level
 327 required to transform the same volume of FCC phase is much lower.

328 The relation between plastic anisotropy, strain path and phase transformation rate, is now
 329 established but still, the observed delay in terms of deformation for $\alpha = 45^\circ$ (see **Fig. 3**) at the
 330 beginning of the transformation is not yet explained or whether a coupling with the rate does
 331 exist.

332



333 **Figure 5:** Evolution of the strain-induced FCC to HCP transformation rate in tension with the
 334 strain path deviation as compared to the isotropic case ($m(\alpha) = a \times |\beta-\beta_{iso}| + b$ with $a = -38 \pm 4$,
 335 $b = 1.63 \pm 0.26$ and $R^2 = 0.99$).
 336

337

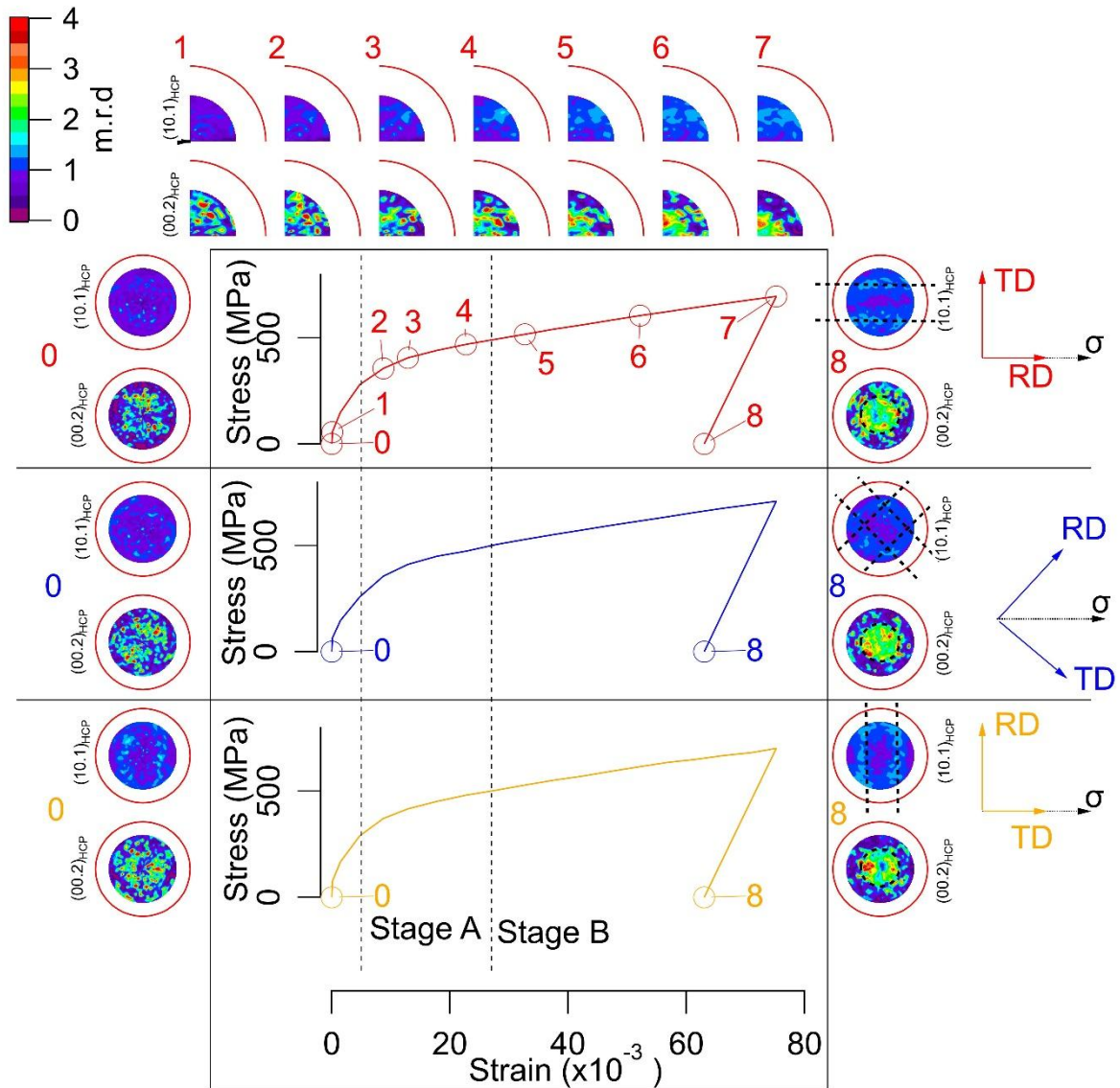
338 3.4. In situ texture evolution, work hardening mechanisms and phase transformation

339 initiation

340 *In situ* crystallographic texture was tracked during tensile tests for each sample (0° /RD (RD),
341 45° /RD and 90° /RD (TD)). **Fig. 6** illustrates $\{00.2\}_{\text{HCP}}$ and $\{10.1\}_{\text{HCP}}$ PFs for some analysed
342 strain levels. The 0 point represents the unstrained state i.e. as received cobalt sheet, points 1
343 to 7 match the texture analyses made at different loading steps, and the point 8 corresponds to
344 the final texture after tensile test. **Fig. 6** also shows macroscopic stress-strain curves obtained
345 by the tensile tests for the 3 sampling directions (0° /RD (RD), 45° /RD and 90° /RD (TD $^\circ$)).
346 Evolution of $\{00.2\}_{\text{HCP}}$ PF with macroscopic strain shows, whatever α , a homogeneization
347 and a reinforcement of the fiber character of the texture. This basal texture results in an
348 equiaxed central pole, representative of the normal c-axis on the specimen surface. The
349 equiaxiality of the pole is representative of cold-rolled HCP materials with a c/a ratio close to
350 1.633. Consequently, the texture that develops during a tensile deformation reveals the
351 reinforcement of the initial rolling texture. Evolution of $\{10.1\}_{\text{HCP}}$ PF with strain shows,
352 whatever α , the development of poles in the rolling direction in accordance with the observed
353 evolution of intensities recorded on $\{00.2\}_{\text{HCP}}$ PF, supporting the assumption of a
354 reinforcement of this type of texture.

355 This qualitative analysis highlights a texture evolution which provides an explanation for the
356 phase transformation activation delay for the 45° /RD tensile loading.

357



358

359 **Figure 6:** $\{00.2\}_{\text{HCP}}$ and $\{10.1\}_{\text{HCP}}$ PF evolution during tensile tests. Points 0 and 8
 360 correspond to the initial texture and the residual texture after straining, respectively. Points 1
 361 to 7 correspond to internal texture development during loading. (red color is associated to
 362 force applied along the RD ($\alpha = 0^\circ$ /RD), blue for an angle of 45° /RD and green for a
 363 mechanical loading parallel to TD ($\alpha = 90^\circ$ /RD)).

364

365 As shown in a previous work [58], the combined study of work-hardening mechanisms and
 366 phase transformation can be done monitoring the intensity of two diffraction reflections. The
 367 $\{00.2\}_{\text{HCP}}$ reflection is representative of the main twinning system $\{10.2\}\langle 10.1 \rangle$, and the
 368 $\{10.2\}_{\text{HCP}}$ reflection, averaged over a ring corresponding to a declination angle of 5° , is
 369 representative of the FCC to HCP phase transformation [7]. For this purpose, the integrated

370 intensities of the $\{00.2\}_{\text{HCP}}$ and $\{10.2\}_{\text{HCP}}$ reflections were tracked during the mechanical
371 loading along the three loading directions (**Fig. 7**).

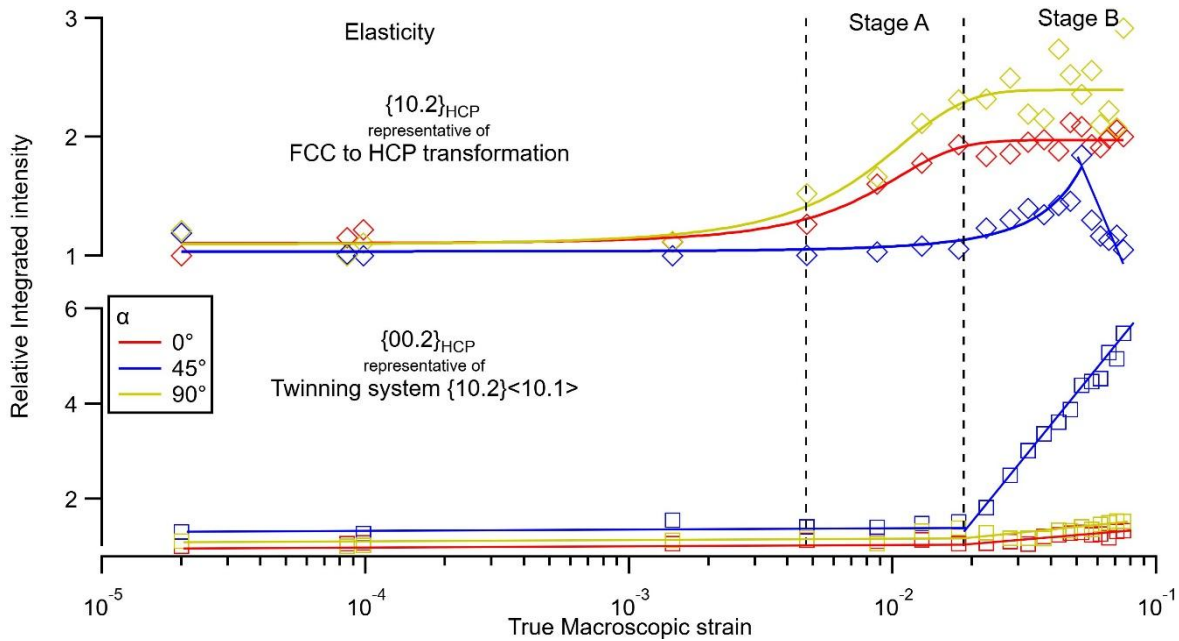
372 No matter the loading direction, intensity of $\{00.2\}_{\text{HCP}}$ reflection starts to increase linearly
373 with the logarithm of the deformation from the beginning of the stage B. This increase reflects
374 the generalization of twinning in this stage and in particular the reorientation due to the
375 $\{10.2\}\langle 10.1 \rangle$ system, since it induces an increase of the $\{00.2\}_{\text{HCP}}$ population parallel to the
376 surface specimen. The effect of plastic anisotropy on the twinning is clear with a very
377 pronounced increase in the intensity of the $\{00.2\}_{\text{HCP}}$ reflection in the case of $\alpha = 45^\circ$ as
378 compared to the other directions which show a similar, but less important, evolution. This
379 may demonstrate an increased twinning activity, favored by the tensile specimen orientation
380 regarding the loading direction.

381 For 0° /RD and 90° / RD, intensity of $\{10.2\}_{\text{HCP}}$ reflection increases at the end of the elastic
382 domain due to the beginning of the FCC to HCP strain-induced transformation. At the
383 beginning of stage B, intensity reaches a plateau. This trend is similar to the observations of
384 Marx *et al.* [42]. However, these authors did not correlate the work-hardening mechanisms
385 and the intensity evolution. As shown in **Fig. 3**, the overall proportion of FCC continues to
386 decrease during this stage. The $\{10.2\}_{\text{HCP}}$ intensity should therefore go on increasing as
387 regard to the phase transformation. Nevertheless, the reorientation due to the activation of the
388 $\{10.2\}\langle 10.1 \rangle$ twinning system tends to decrease the $\{10.2\}_{\text{HCP}}$ intensity. The stabilization
389 observed is therefore the result of the two competing mechanisms.

390 For $\alpha = 45^\circ$ /RD, the delayed initiation of the FCC to HCP transformation, identified in **Fig. 3**,
391 is also visible in **Fig. 7** through a significant increase in integrated intensity of $\{10.2\}_{\text{HCP}}$ peak
392 at the beginning of stage B. However, in the middle of the stage B, the effect of the twinning
393 mode deformation onto intensity overrides those of the phase transformation, leading to a

394 sudden drop in intensity. The initial basal texture is not favorable to the $\{00.1\}\langle 1\bar{2}.0\rangle$ slip
 395 system activation when the sheet is deformed at $45^\circ/\text{RD}$.

396



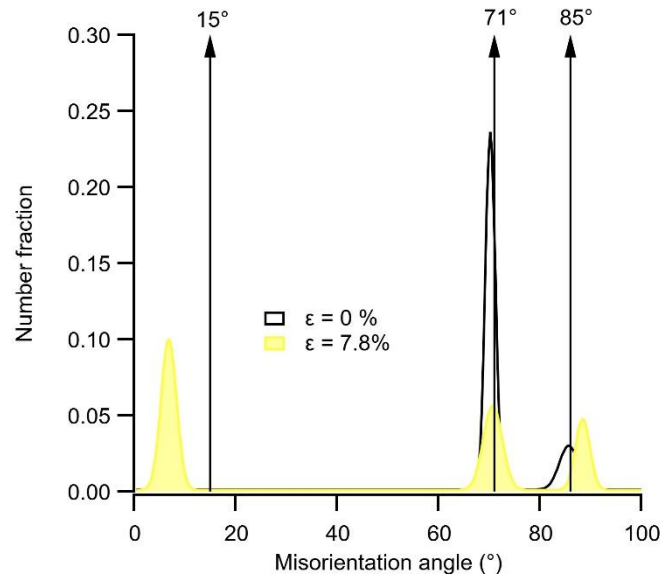
397

398 **Figure 7:** Evolution of the integrated peak intensities of the $\{10.2\}_{\text{HCP}}$ reflection (at $\psi = 0^\circ$)
 399 and of the $\{00.2\}_{\text{HCP}}$ reflection (at $\psi = 5^\circ$) during a tensile test (ψ is the declination angle) for
 400 all three directions of specimen collection.
 401

402 Additional *post mortem* analysis of the work-hardening mechanisms was carried out by EBSD
 403 in the case of the tensile specimen at $0^\circ/\text{RD}$. A quantification of the grain misorientation
 404 angles is presented in **Fig. 8** thanks to the orientation analysis of their distribution in the
 405 undeformed and deformed states. The as-received cobalt sheet exhibits only two set of
 406 populations : the first one displays a misorientation angle corresponding to the Shoji-
 407 Nishiyama orientation relationship (71°) between FCC and HCP phases, associated to the
 408 martensitic transformation [10], while the second one manifests a 86° misorientation angle,
 409 typical of the initial existence of residual tensile twins $\{10.2\}\langle 10.1\rangle$ due to the cold-rolling
 410 process. Above 7.8 % of macroscopic deformation, the twin population increases while the
 411 peak located at 71° decreases, highlighting the two mechanisms of twinning and strain-

412 induced phase transformation (FCC to HCP). A third population appears for misorientation
413 angles lower than 15° . It may be associated with the development of intragranular
414 misorientations due to dislocations and internal stresses [7].

415



416

417 **Figure 8:** Grain boundary misorientation angle distributions for unstrained state (black solid
418 line) and for a specimen strained in tension at 7.8 % along RD (yellow filled curve).

419

420 4. Conclusion

421 In this work, the strain-induced phase transformation is reported for three sampling directions
422 of a cold-rolled sheet of pure cobalt and for a complex loading which produces higher strain
423 levels than those obtained with tensile tests. The effect of anisotropy is demonstrated to be
424 important for a loading axis at 45° from the RD, with a delayed activation of the FCC to HCP
425 phase transformation and a rate of transformation twice as high as for the rolling and
426 transverse directions. This second observation can be associated with a negative deviation of
427 the strain path as regard to the isotropic case. Moreover, this effect seems to favor twinning
428 deformation mode that ends up transcending phase transformation. For RD (0° /RD) and TD

429 (90° /RD) directions, whatever the loading mode (tensile or stamping tests), the phase
430 transformation starts at the initiation of plasticity (stage A). In general, the deformation of
431 polycrystalline cobalt is controlled by basal slip and twinning (mainly {10.2}<10.1> tensile
432 twin) but can be accompanied by a plastic strain-induced transformation if the residual FCC
433 phase is not stabilized by a heat treatment. The texture developed during deformation is then a
434 reinforcement of the basal rolling texture.

435

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441

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608

610 **Figures captions:**

611

612 **Figure 1:** Microstructural analysis of the as-received material: (a) EBSD map of phase
613 distribution and band contrast. Distribution of grain size for each phase. HCP phase is colored
614 in red and FCC phase in green. (b) XRD crystallographic textures of the HCP phase
615 determined from the orientation distribution functions calculated by the WIMW method
616 implanted in the BEARTEX software.

617

618 **Figure 2:** Description of the Nakazima test: (a) height of the part after stamping and location
619 of the analysis points by XRD (cross and circle marker for measurements in RD and TD,
620 respectively), (b) decomposition of the principal components of the macroscopic strain fields
621 (ϵ_I : in-plane major strain (along RD), ϵ_{II} : in-plane minor strain (along TD), $\epsilon_{III}=-\epsilon_I-$ ϵ_{II} : in the
622 thickness strain and corresponding ϵ_{eq} von Mises equivalent strain), (c) evolution of the strain
623 path β versus the location on the section.

624

625 **Figure 3:** FCC phase proportion (normalized to the initial retained FCC phase) and work-
626 hardening rate evolutions under tensile testing for different angles between the load axis and
627 the rolling direction (RD): 0° /RD (RD), 45° /RD and 90° /RD (TD $^\circ$).

628

629 **Figure 4:** Influence of different biaxial loading scenarios on the phase transformation.
630 Evolution of the FCC phase ratio along the rolling and transverse directions (filled squares) of
631 a stamped sample, superposed on the in situ tensile test results (circles).

632

633 **Figure 5:** Evolution of the strain-induced FCC to HCP transformation rate in tension with the
634 strain path deviation as compared to the isotropic case ($m(\alpha) = a \times |\beta - \beta_{iso}| + b$ with $a = -38 \pm 4$,
635 $b = 1.63 \pm 0.26$ and $R^2 = 0.99$).

636

637 **Figure 6:** $\{00.2\}_{HCP}$ and $\{10.1\}_{HCP}$ PF evolution during tensile tests. Points 0 and 8
638 correspond to the initial texture and the residual texture after straining, respectively. Points 1
639 to 7 correspond to internal texture development during loading. (red color is associated to
640 force applied along the RD ($\alpha = 0^\circ$ /RD), blue for an angle of 45° /RD and green for a
641 mechanical loading parallel to TD ($\alpha = 90^\circ$ /RD)).

642

643 **Figure 7:** Evolution of the integrated peak intensities of the $\{10.2\}_{HCP}$ reflection (at $\psi = 0^\circ$)
644 and of the $\{00.2\}_{HCP}$ reflection (at $\psi = 5^\circ$) during a tensile test (ψ is the declination angle) for
645 all three directions of specimen collection.

646

647 **Figure 8:** Grain boundary misorientation angle distributions for unstrained state (black solid
648 line) and for a specimen strained in tension at 7.8 % along RD (yellow filled curve).

649

650 **Tables captions:**

651

652 **Table 1:** Review of the main coefficients characterizing elastic and plastic anisotropies
653 determined for polycrystalline specimens (E: Young modulus, ν : Poisson coefficient,
654 α : direction of specimen collection, r: Lankford coefficient, β : strain path, $|\beta - \beta_{\text{iso}}|$: strain path
655 deviation, r_{mean} : mean plastic anisotropy coefficient, Δr : planar plastic anisotropy coefficient).
656

657 **Table 2:** Coefficients of equation (8) with standard deviations (u) and linear regression
658 coefficient (R^2) as a function of the loading axis misorientation regarding the RD.

659

660

661