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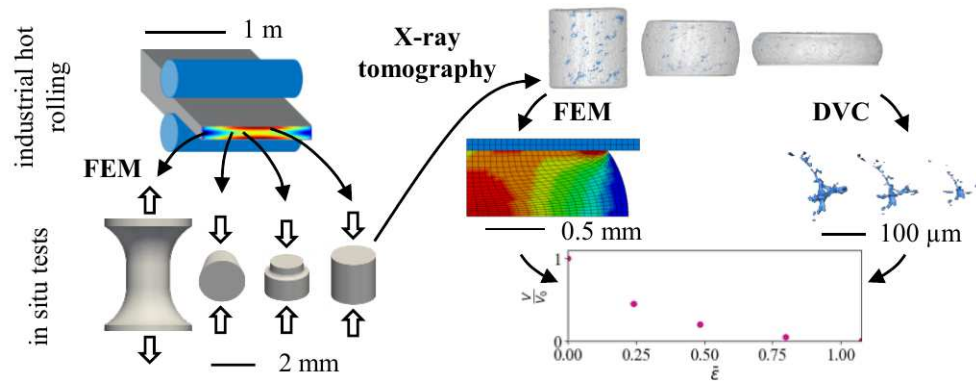
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Graphical Abstract

**Pore closure in thick aluminum plate:
from industrial hot rolling to individual pore observation**

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Pore closure in thick aluminum plate: from industrial hot rolling to individual pore observation

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Abstract

Pores are often present in large aluminum ingots after casting. To ensure the mechanical reliability of the final thick plates, these pores must be closed during the forming process, hot rolling in the present case. This study aims at understanding the effects of rolling parameters on the volume evolution of pores. To do so, X-ray microtomography is used to track real casting pores during deformation. Nevertheless, thick plates are too large to enable a fine characterization of the evolution of pores during the process. The size and shape of the samples as well as the mechanical boundary conditions must be optimized to meet imaging constraints. This paper focuses on the reproduction of complex loading paths. Multi-scale FE simulations are used to reproduce the loading conditions of thick plate rolling with uniaxial tests on samples of a few millimeters. Uniaxial tests are then characterized with tomography on a synchrotron X-ray beamline. In each sample, tens of pores are individually tracked, giving access to their volume evolution. The local loading path experienced by each pore is determined with FE simulation. It is shown that the volume evolution of a real pore correlates with the hydrostatic integration which is the integral of stress triaxiality along cumulated strain. This confirms that

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the closure of complex casting pores is enhanced by a high relative reduction and high values of L/H ratio during rolling similarly to what was observed numerically on simpler shapes.

Keywords: Aluminum, Hot rolling, FE simulation, in situ X-ray microtomography, Pore volume evolution

1. Introduction

Aluminum alloys are largely used in aerospace manufacturing thanks to their low density, their good mechanical properties, and their high resistance to corrosion. Their use as thick plate in aerospace applications depends on the mastery of processing parameters. It is necessary to avoid the presence of pores in the final product and the concomitant deterioration of mechanical properties. Industrially, the soundness of the material is ensured by control tests such as dye penetrant inspection. The number of pores, their size and the distance between pores must be in a certain range. Pores in aluminum products appear during casting. They are caused by the increase of density and the decrease of hydrogen solubility at solidification. In the case of thick aluminum plate, pores are closed by plastic deformation during hot rolling. The optimization of this process is nevertheless complex. There is a large number of relevant rolling parameters (temperature, reduction per pass, roll radius, roll velocity) and their dependence on each other precludes a systematic experimental campaign at the scale of industrial rolling mill. It is essential to accurately describe the closure of pores to ensure the final soundness of the plate.

Park and Yang (1996) showed that pore closure occurs in two steps. First morphology and topology of the pore evolve, along with the pore volume decrease. Then, the internal surfaces of the pore weld together during bounding. In the following, we will only focus on the first step also called mechanical closure. Llanos et al. (2008) investigated pore closure during rolling by using 2D post mortem observations. Such approach allows real pores to be investigated but does not give access to volume evolution. Chen et al. (2011) and later Joo

25 et al. (2014) used artificial drilled pores in order to track the pore volume evolu-
26 tion during rolling. They found that pore closure is influenced by the reduction
27 of the pass, the pore size and the diameter of the rolls. However, it has been
28 shown numerically by Hwang and Chen (2002) in 2D and by Chen et al. (2010)
29 in 3D that pore shape has a significant effect on pore closure kinetics. Since real
30 pore surrounded by matter cannot be considered as drilled pore, Youssef et al.
31 (2006) and Toda et al. (2009) performed 3D observations of real pores. On the
32 one hand, real complex pores were observed but they were not tracked along
33 the rolling pass schedule. On the other hand, same pores were observed before
34 and after hot rolling. However, in this latter case, only spherical micropores
35 were studied whereas pores in aluminum alloys can have branched and tortuous
36 shapes. Characterization of real complex pores during their closure will allow
37 the understanding of their closure mechanisms and closure kinetics to improve.
38 Then, the current models of pore closure can be validated or improved .

39
40 X-ray microtomography offers both a 3D and non destructive characteriza-
41 tion at a spatial resolution (order of magnitude of micrometer) compatible with
42 pore description. The non destructive aspect allows several 3D images to be
43 captured per pore during deformation. To save time, sample size was limited
44 to several millimeters. In this way, the whole sample and pores are imaged
45 with sufficient resolution and a unique scan. Such a sample contains several
46 tens of pores, providing many pore volume evolutions to investigate closure ki-
47 netics. Samples are deformed at high temperature using a cumbersome setup
48 that requires a synchrotron X-ray source to perform interrupted in situ tests.
49 Consequently, there is significant difference of size between, on the one hand,
50 industrial plate ingots that are hot rolled, which size is about $4\text{m} \times 1.5\text{m} \times$
51 0.5m , and on the other hand, samples of several millimeters in which pores can
52 be observed. It is necessary to ensure that the loading path experienced by
53 pores during hot rolling can be reproduced with the compression of samples
54 compatible with X-ray microtomography.

55

56 In the following, a combination of numerical and experimental approaches
57 is presented. First, finite element simulations are used to compare the load-
58 ing paths encountered during industrial thick plate rolling to the loading paths
59 encountered during uniaxial tests of small samples with varied shapes. A ded-
60 icated experimental setup has been developed to perform high temperature in
61 situ tests characterized by X-ray microtomography. The volume evolution of
62 pores during their deformation are determined using 3D quantitative analysis
63 methods.

64 **2. Numerical and experimental methods**

65 *2.1. Numerical simulations*

66 Rolling of industrial plate and a uniaxial mechanical test on a sample com-
67 patible with X-ray microtomography differ in terms of sample size and nature
68 of loading. Finite element (FE) simulations were used to ensure that uniaxial
69 tests are representative of the loading states reached with rolling.

70

71 Hot rolling simulations are run with LAM3 software (Hacquín, 1996). The
72 model accounts for thermal and mechanical phenomena involved in rolling (such
73 as the deformation of the plate and the rolls, the thermal exchanges between the
74 rolls and the plate, between the plate and the air and the temperature increase
75 in the plate due to mechanical deformation). It couples finite element method
76 to compute the elasto-viscoplastic behavior of the plate and semi-analytic com-
77 putations of the deformation of the rolls. A stationary Eulerian formulation is
78 used, thus the transient regime at both ends of the plate are not accessible. A
79 classical rolling pass schedule for thick plates requires a progressive reduction
80 of the plate. Several passes between the rolls are necessary to reach the desired
81 final thickness. In the following, we will study the reproduction of the load-
82 ing state of individual passes. Each pass geometry is usually described by two
83 parameters which are the relative reduction r (Equation 1) and the L/H ratio
84 (Equation 2), where L is the contact length between the rolls and the plate

85 and H is the mean thickness of the plate. h_1 and h_2 are the entry and exit
 86 thicknesses respectively and R_{cyl} is the radius of the rolls (Figure 1).

$$r = \frac{h_1 - h_2}{h_1} \in [0, 1] \quad (1)$$

$$\frac{L}{H} = \frac{2\sqrt{R_{cyl}(h_1 - h_2)}}{h_1 + h_2} \quad (2)$$

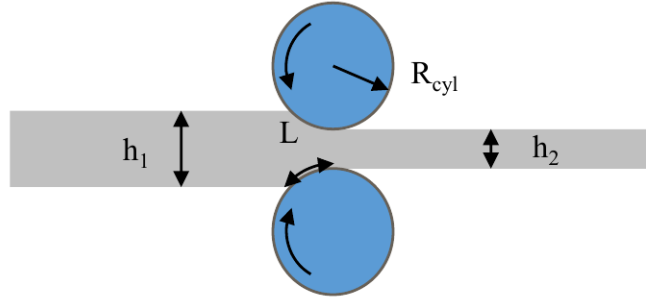


Figure 1: Schematic description of a rolling pass. h_1 : entry thickness, h_2 : exit thickness, R_{cyl} : radius of the rolls, L : contact length between the rolls and the plate.

87 FE simulations of uniaxial tests are performed using the software LS-DYNA (LS-
 88 DYNA, 2017). Different sample geometries are tested either in compression
 89 (cylinder, Brazilian and double-collar) or in tension to broaden the range of
 90 accessible loading states (Figure 2). Double-collar geometry was inspired from

91 the work of Farrugia (2006). For cylinders, double-collars and tension samples,
 92 2D axisymmetric elements are used. For Brazilian tests, 3D hexahedral fully
 93 integrated elements are used. The material constitutive equations follow the
 94 Sellars-Tegart law (Equation 3) (Sellars and Tegart, 1972).

$$\sigma = \frac{1}{\alpha} \ln \left(\left(\frac{Z}{A} \right)^{\frac{1}{m}} + \sqrt{\left(\frac{Z}{A} \right)^{\frac{2}{m}} + 1} \right), \quad Z = \dot{\epsilon} \exp \left(\frac{Q}{RT} \right) \quad (3)$$

95 The four parameters α , A , m and Q were determined experimentally using
 96 the method described by Zhu et al. (2018). Most of the time, the pores are stud-
 97 ied in a matrix following a Norton power law of this type $\dot{\epsilon} = A \sigma^n$. For a given
 98 temperature of 480 °C, the Norton index n of the present material is of about
 99 6. Usually, the Norton index of aluminum alloys is between 3 and 10, which
 100 limits its effect on pore closure reported by Zhang et al. (2009). Besides, Saby
 101 et al. (2015a) observed that the effect of the Norton exponent in this range is
 102 negligible compared to the effect of the orientation or the shape of the pore. In
 103 the simulation software, the material law consists in several stress-strain curves
 104 at different temperatures and strain rates: 10 temperatures between 380 and
 105 580 °C and 10 strain rates between 1×10^{-3} and $1 \times 10^2 \text{ s}^{-1}$. For compression
 106 of cylinders, Brazilians and double-collars, infinitely rigid dies are used in the
 107 simulation. A coefficient of static friction of 0.4 between the die and the sample
 108 is applied. This coefficient has been chosen in order to match barreling effect
 109 that is observed experimentally during compression test. Concerning thermal
 110 conditions, the initial temperature is fixed at all sample nodes and no exchanges
 111 are considered between the dies and the air. The simulation takes into account
 112 the heat due to the mechanical work. Compression tests were simulated up to
 113 a relative reduction of 60% and tensile tests up to initiation of necking.

114

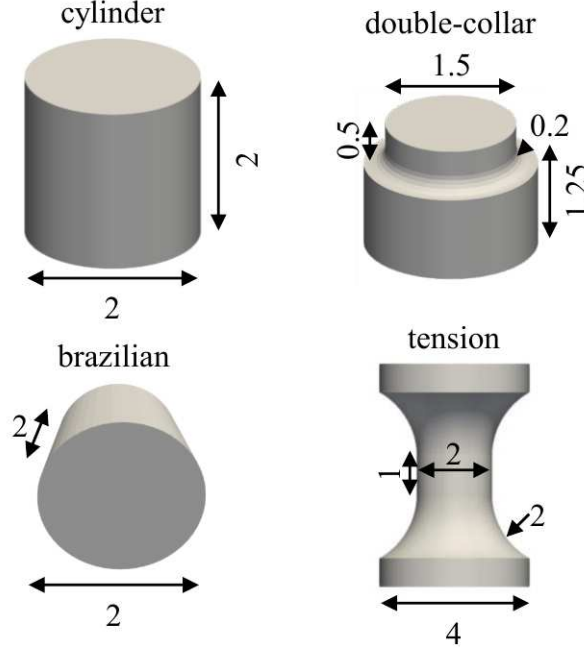


Figure 2: Simulated geometries to investigate loading paths under uniaxial loading. Dimensions are given in millimeters. Loading direction is vertical.

115 The dimension of pores are small compared to the sample size. It is assumed
 116 that the mechanical fields are not modified by the presence of the pores. There-
 117 fore, the simulations were performed on samples free of pores. In the literature,
 118 cumulated strain (Equation 4) and stress triaxiality (Equation 5) are shown to
 119 be the two main mechanical parameters involved in pore closure. They are used
 120 in the analytical models of Gurson (1977) and Duva and Hutchinson (1984), the
 121 semi-analytical model of Zhang et al. (2009) and the phenomenological model
 122 of Saby et al. (2015a). Thus, loading states will first be compared in terms of
 123 triaxiality T_X and cumulated strain $\bar{\epsilon}$.

$$\bar{\epsilon} = \int_0^t \dot{\bar{\epsilon}} dt \quad \dot{\bar{\epsilon}} = \sqrt{\frac{2}{3} \frac{\dot{\epsilon}_{ij} \dot{\epsilon}_{ij}}{\dot{\epsilon}_{kk}}} = \frac{\partial \bar{\epsilon}}{\partial t} \quad (4)$$

$$T_X = -\frac{p}{\bar{\sigma}} \quad p = -\frac{1}{3} \operatorname{tr}(\underline{\underline{\sigma}}) \quad \bar{\sigma} = \sqrt{\frac{3}{2} \underline{\underline{\sigma}}' : \underline{\underline{\sigma}}'} \quad \underline{\underline{\sigma}}' = \underline{\underline{\sigma}} + p\underline{\underline{I}} \quad (5)$$

124 Where $\underline{\underline{\varepsilon}}$ is the strain tensor, $\underline{\underline{\sigma}}$ the stress tensor and p the hydrostatic pres-
 125 sure.

126 The stress state is more precisely represented considering the Lode parameter
 127 μ (Equation 6) in addition to the stress triaxiality.

$$\mu = \frac{2\sigma_2 - \sigma_1 - \sigma_3}{\sigma_1 - \sigma_3} \quad (6)$$

128 The Lode parameter influence has been mostly studied in the scope of failure
 129 of ductile metals. It has been observed by Danas and Ponte Castañeda (2012)
 130 that at high triaxiality, ductile failure is controlled by triaxiality but when triax-
 131 iality decreases (in absolute value), the Lode parameter plays a significant role.
 132 (Zhang et al., 2001) demonstrated that, at low positive triaxiality, the Lode pa-
 133 rameter has a non-negligible influence on the pore shape rather than on the pore
 134 volume. Chbihi et al. (2017) studied pore closure using FE simulations, they
 135 concluded that at low triaxiality, high values of the Lode parameter foster pore
 136 closure. More recently, Zhang et al. (2020) and Wang and Dong (2020) proposed
 137 two new prediction models of pore closure that incorporate the Lode parameter.

138

139 2.2. Experimental data acquisition

140 A dedicated micropress has been developed (Figure 3.b) to perform in situ
 141 mechanical tests at high temperature. The device can perform compression
 142 and tensile tests at a controlled speed (up to 1 mm.s^{-1}). The applied load is
 143 recorded. The sample is placed between the movable piston and the fixed bell
 144 (made of boron nitride to be transparent to X-rays) (Figure 3.c). The micro-
 145 press is mounted on the rotating stage and a furnace is moved down around the
 146 bell to heat the sample (Figure 3.a). The furnace is independent from the ro-
 147 tation stage and does not move during the tomography acquisition. Two glassy
 148 carbon windows are located on the X-ray path at the entrance and exit of the

149 furnace.

150

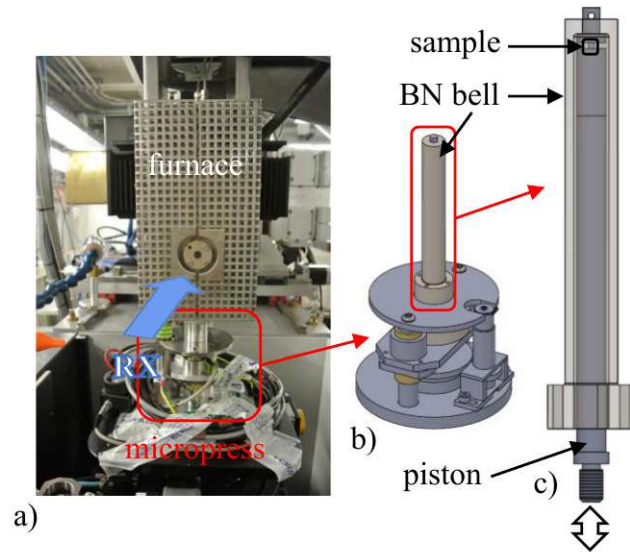


Figure 3: Experimental setup: a) picture of the micropress and the furnace on the ESRF-ID19 beamline. b) Sketch of the micropress. c) Sketch of the upper part of the micropress composed of the bell and the piston surrounding the sample.

151 In situ tests at high temperature are performed with a synchrotron X-
152 ray source, at the ESRF (European Synchrotron Radiation Facility, Grenoble,
153 France) on ID19 beamline. It provides a parallel quasi-monochromatic coherent
154 X-ray beam with a high flux of photons. A 34 keV X-ray beam energy was used
155 providing projections with a pixel size of $2.44 \mu m$.

156 To reproduce real rolling conditions, a high piston velocity is used ($1 mm.s^{-1}$).
157 It ensures a strain rate of about $1 s^{-1}$. Thus, interrupted in situ tests are
158 performed (Figure 4) with fast tomography acquisition (around 10s for a full
159 scan) to avoid any relaxation problem that might blur images. For each scan,

160 2000 radiographs of 1536×1440 pixels are recorded using a PCO edge detector
 161 mounted on a $\times 5$ optic with a $100 \mu\text{m}$ thick YAG scintillator. Reconstructions
 162 are performed with the software PyHST (High Speed Tomography in python
 163 version) (Mirone et al., 2014) using a phase contrast resolution with an approach
 164 developed by Paganin et al. (2002) and filtered backprojection. Ring artifacts
 165 are removed with an in house ESRF Matlab script on reconstructed volumes
 166 (Lyckegaard et al., 2011).

167

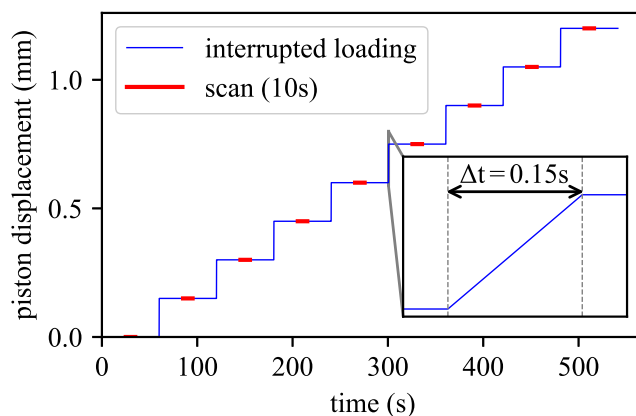


Figure 4: Displacement of the die as a function of time for an interrupted compression in 8 steps of $150 \mu\text{m}$ at 1 mm.s^{-1} .

168 A total of 18 samples have been tested with different geometries (11 cylin-
 169 ders, 4 double-collars, 2 Brazilians and 1 tensile test). For cylinders, different
 170 temperature (from 430 to 520°C), compression speeds (from 0.1 to 1 mm.s^{-1})
 171 and alloys have been tested (2XXX and 7XXX series).

172 2.3. Data processing and tracking method

173 Three phases are distinguishable in the tomographic data: pores, matrix
 174 and intermetallics (as visible in Figure 5). Image processing involving filters
 175 and thresholding is used to isolate the pores (with ImageJ software (Schneider
 176 et al., 2012)). Two consecutive backgrounds were subtracted from the image.

177 The first one is generated using a 2D mean filter with a radius of 20 voxels.
178 It allows pores to be detected and to fill them to generate a second 2D mean
179 background with a radius of 10 voxels. Then pore thresholding is performed
180 using a cutoff value kept constant for all the samples. In the following, only
181 pores with a volume higher than 8 voxels (or $116 \mu m^3$) will be considered.

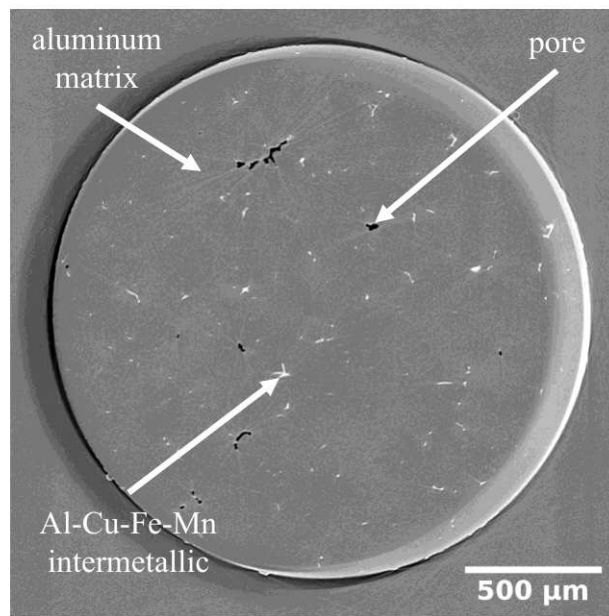


Figure 5: Visible phases in a cross section of a cylindrical sample after reconstruction: pores (dark), intermetallics (bright) and the matrix (medium grey).

182 The interest of in situ tests is to follow the morphological evolution of pores
183 during sample deformation. However, pores tracking from one scan to another
184 is not straightforward due to the large number of pores (several tens), the large
185 and heterogeneous deformation of the sample and the topological evolution of
186 pores (fragmentation, change of shape). Hence, an automatic procedure has
187 been developed by Lhuissier et al. (2021) to track pores all along the deforma-

188 tion. This procedure based on Digital Volume Correlation (DVC), provides an
189 estimate of the displacement field thanks to the tracking of intermetallics. The
190 interpolation of the displacement field allows to track pores.

191 The local mechanical field is obtained thanks to FE numerical simulation
192 with LS-DYNA. The boundary conditions applied as well as the sample shape
193 for the simulation are based on the in situ experiments: real sample shape is
194 used and measured displacement of dies is applied.

195 All in all, 18 samples have been scanned with a total of 158 scans. The
196 data processing workflow has been automated to get reliable and comparable
197 data from one scan to another (Figure 6). Image processing and DVC give
198 access to the morphological evolution of every pore in every sample during the
199 compression or tensile tests. Coupling with simulation, which provides local
200 loading path, it is possible to access individual pore closure or opening kinetics,
201 meaning the relationship between morphological evolution and local loading
202 conditions. An example of such evolution is given in Figure 6 with the pore
203 volume change as a function of cumulated strain.

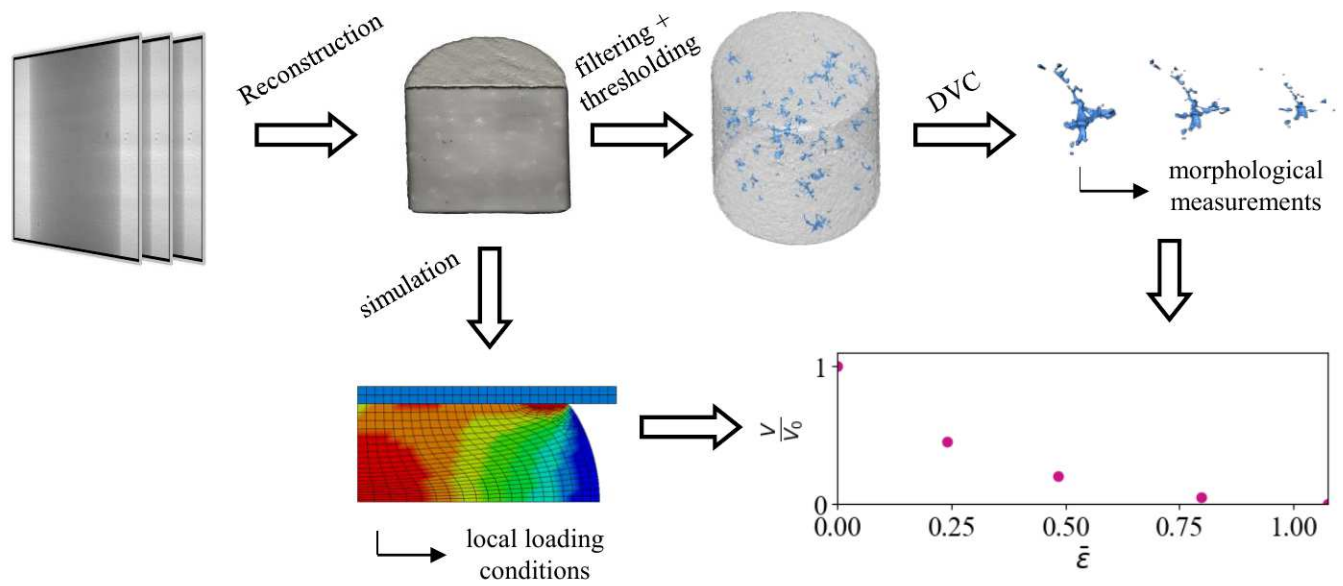


Figure 6: Processing workflow to obtain the pore closure kinetics from radiographs acquired with tomography.

204 **3. Results and discussions**

205 *3.1. Loading paths during hot rolling*

206 During hot rolling, thermo-mechanical fields are not homogeneous in the
207 transverse section of the plate (section perpendicular to the rolling direction).
208 Figure 7 presents the distribution of hydrostatic pressure within the section
209 located right between the rolls. The thickness is the dimension of the plate
210 along Short-Transverse (ST) direction. The width is along Long-Transverse
211 (LT) direction. High hydrostatic pressure is reached close to the rolls at the mid-
212 width while low hydrostatic pressure is reached on the edges at mid-thickness.
213 In this area of negative hydrostatic pressure, pores will tend to open. Thus,
214 loading path is highly dependent on the considered position in the transverse
215 section.

216 Mechanical fields at five representative positions in the plate are studied
217 (positions marked on Figure 7). The *center* position is located at mid-width and
218 mid-thickness. Two other positions are studied along mid-width: the *surface*
219 position which is in contact with the rolls and the *quarter thickness* position
220 which is initially (i.e. before rolling) at mid-way between *center* and *surface*
221 positions. Along mid-thickness, *extreme edge* position is at the side surface of
222 the plate and *edge* position is where the hydrostatic pressure reaches lowest
223 values during rolling.

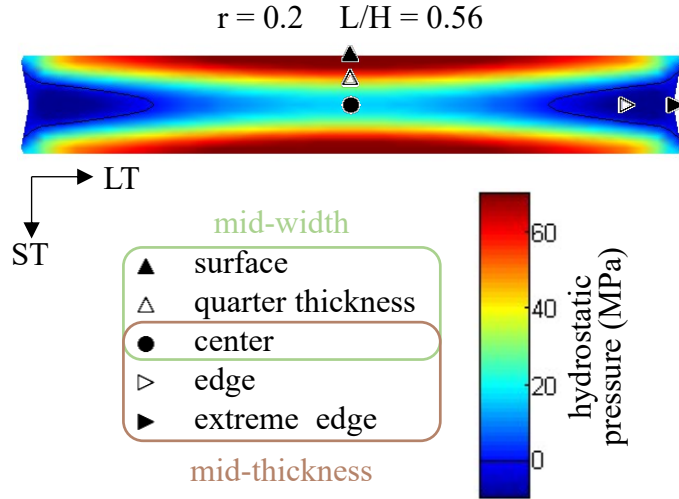


Figure 7: Distribution of hydrostatic pressure within the transverse section between the rolls.

224 Several rolling pass schedules are simulated changing: the reduction per
 225 pass, the roll radii, the temperature, and the roll speed. Each pass is studied
 226 individually, independently of the previous or following passes. Loading paths
 227 are defined as the local evolution of stress triaxiality as a function of the cu-
 228 mulated strain. Figure 8.a shows, for a given pass ($r=0.2$ and $L/H=0.56$), the
 229 different loading paths depending on the position in the plate. Close to the
 230 edges (*edge* and *extreme edge*), tensile state, with positive triaxiality, appears.
 231 At mid-width (*center* and *quarter thickness*) loading state is more compressive
 232 and strain increases towards the rolls (*quarter thickness*). Regardless to the po-
 233 sition, the triaxiality as a function of the cumulated strain is almost constant.
 234 Loading paths can be approximated as the final loading state using two descrip-
 235 tors: the final cumulated strain $\bar{\epsilon}_f$ (i.e. cumulated strain reached locally at the
 236 end of the pass) and the average triaxiality $\langle T_X \rangle_f$ (Equation 7).

$$\langle T_X \rangle_f = \frac{1}{\bar{\epsilon}_f} \int_0^{\bar{\epsilon}_f} T_X \, d\bar{\epsilon} \quad (7)$$

237 Each considered position in the plate has an influence on both $\bar{\epsilon}_f$ and $\langle T_X \rangle_f$.
 238 For a given position, an increase of relative reduction r results in an increase of

239 final cumulated strain (Figure 8.b). The triaxiality is directly linked to the L/H
240 ratio. Higher values of L/H lead to more compressive states (Figure 8.c). These
241 results are consistent with the observations of Wallerö (1985) who recommends
242 large values of relative reduction and L/H ratio to favor pore closure. No effect
243 of rolling temperature nor roll speed was found on the loading states.

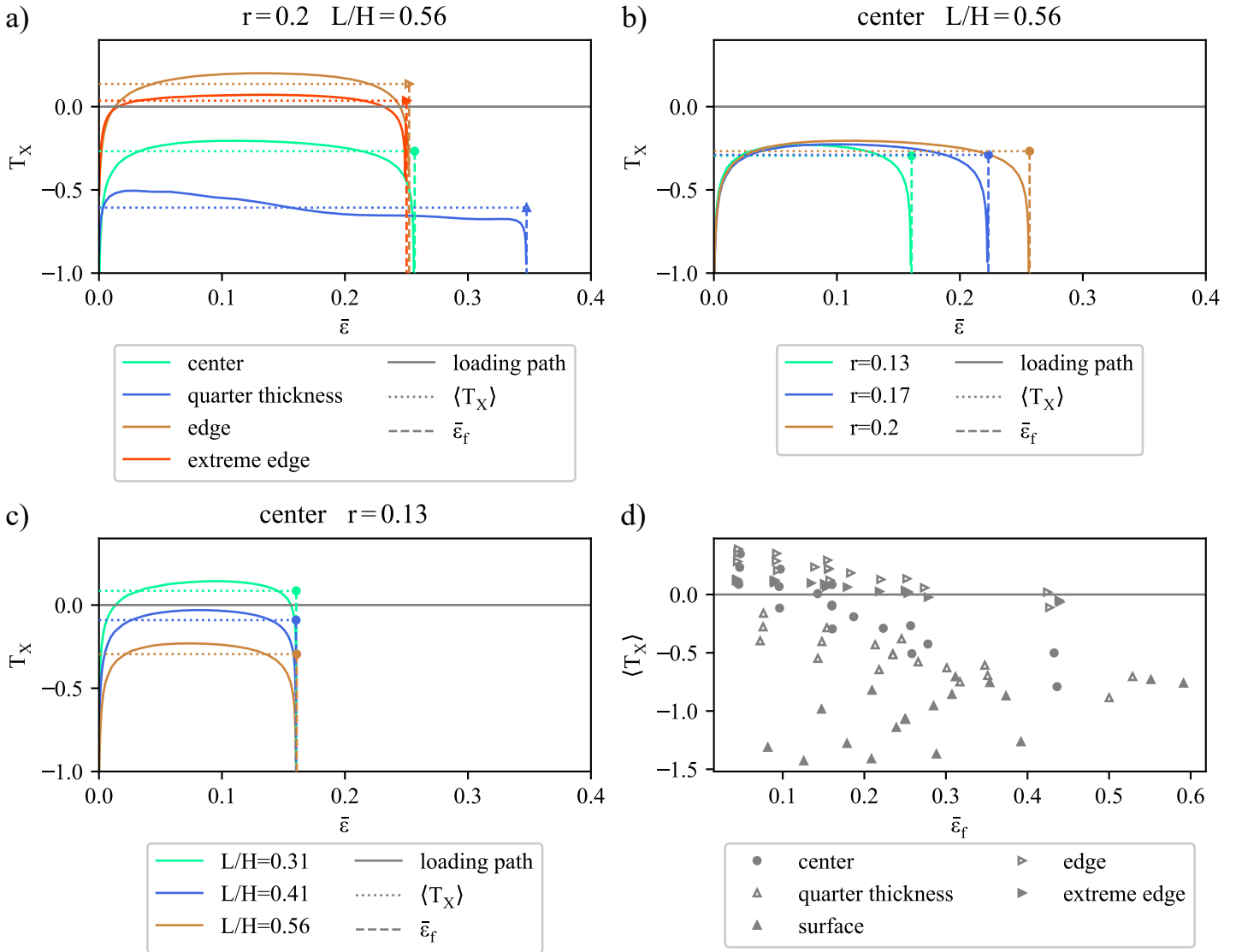


Figure 8: a-c) Effect of different parameters on the loading paths of a single rolling pass: a) position in the transverse section, b) relative reduction value, c) L/H ratio value. d) Loading states of rolling to be reproduced.

244 Figure 8.d shows the loading states at different positions for passes of dif-
 245 ferent pass schedules that could be used for industrial hot rolling. It defines a

246 map of loading states that have to be reproduced with uniaxial tests. Three
247 zones can be distinguished when focusing on the triaxiality: i) the edge, the ex-
248 tremite edge and the center for low deformation where $\langle T_X \rangle \geq 0$; ii) the quarter
249 thickness and the center for large deformation where $0 > \langle T_X \rangle \geq -0.7$; iii) the
250 surface of the plate where $\langle T_X \rangle < -0.7$.

251 Similarly to the stress triaxiality, the Lode parameter along cumulated strain
252 is almost constant (Figure 9.a). For each pass, the Lode parameter path can
253 be locally approximated by the mean Lode parameter and the final cumulated
254 strain. Figure 9.b shows the results for a pass schedule of 5 passes.

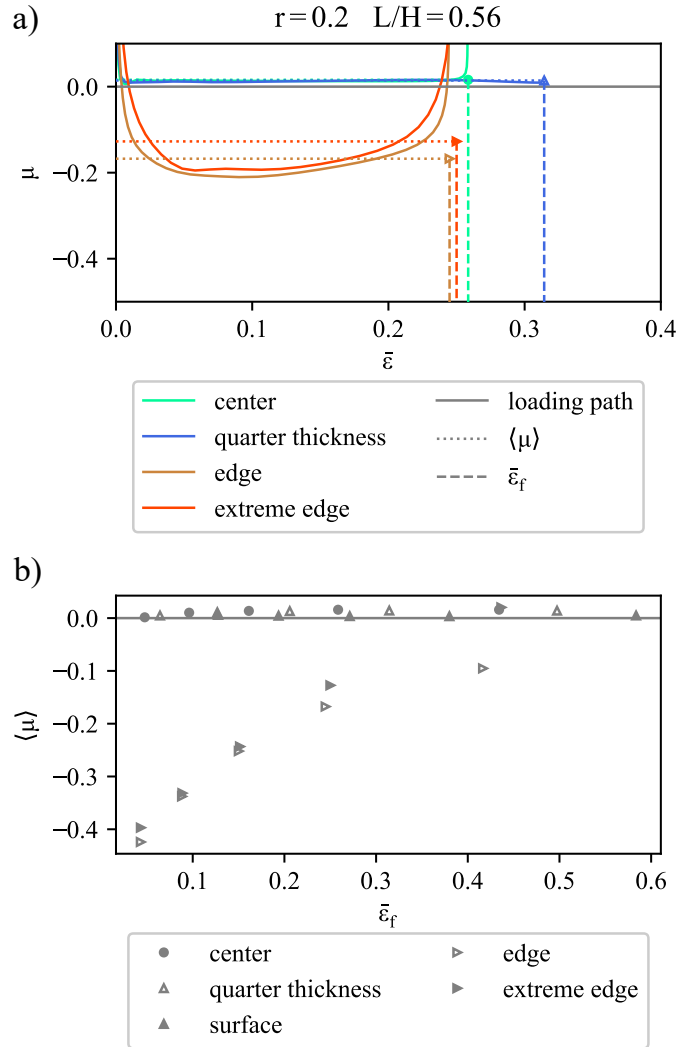


Figure 9: a) Effect of the position on the Lode parameter path b) Lode parameter values obtained for 5 different passes of rolling at different positions in the plate.

255 When looking at the Lode parameter, two zones are distinguished. Away
 256 from the edge, the Lode parameter is zero. This value results from the plane
 257 stain state. Thus, in most of the plate variations of pore closure kinetics are
 258 not influenced by the Lode parameter which is constant. Close to the edges, the

259 plane strain state is not fulfilled and the Lode parameter takes negative values.
 260 Its absolute value decreases when the cumulated strain increases. Besides, these
 261 zones experience low triaxiality (in absolute value). In that case, either in ten-
 262 sion or in compression, Zhang et al. (2001) and Wang and Dong (2020) observed
 263 that the Lode parameter has an influence on the pore volume evolution. They
 264 also observed that the Lode parameter affects the evolution of the pore shape
 265 when observing spherical or ellipsoidal pores. However, in our present case, we
 266 want to study real casting pores which have complex shapes. Most of them are
 267 tortuous and branched. Contrary to ellipsoid, the shape of a real pore cannot
 268 be described with a few number of parameters for which we can determine the
 269 sensibility to the Lode parameter. Thus, the evolution of volume due to the
 270 modification of the pore shape will depend on the Lode parameter value but
 271 also on the initial shape of the pore.

272

273 Considering both stress triaxiality and Lode parameter to describe the stress
 274 states encountered during rolling, we can identify three different zones:

- 275 • The edge of the plate (edge and extreme edge positions) and the center of
 276 the plate for low deformation where $\langle T_X \rangle \geq 0$ and $\langle \mu \rangle \leq 0$.
- 277 • The center of the plate for high deformation and the quarter thickness
 278 where $0 > \langle T_X \rangle \geq -0.7$ and $\langle \mu \rangle = 0$.
- 279 • The surface where $\langle T_X \rangle < -0.7$ and $\langle \mu \rangle = 0$.

280 3.2. Loading paths during *in situ* tests

281 Simulations are performed to determine the loading states accessible with
 282 uniaxial mechanical tests on small samples. The objective is to find sample
 283 geometries that cover the entire map of loading states previously defined (Fig-
 284 ure 8.d).

285 The loading states of uniaxial tests can be compared to those of rolling using
 286 the mean triaxiality and the cumulated strain at the end of deformation. How-
 287 ever, it is not relevant to do so since the final cumulated strain only depends

288 on the total displacement of the dies imposed during the test. For example, in
 289 compression (Figure 10.a), at the center of the cylinder, the final strain reaches
 290 a higher value than for a single rolling pass (2 versus 1.2). Nevertheless, all
 291 intermediate cumulated strain values up to the final value give access to many
 292 mean triaxiality ratios because triaxiality is not constant during deformation
 293 for uniaxial tests. Thus, for each sample, the accessible loading states are repre-
 294 sented by the envelope of all the loading paths $\langle T_X \rangle = f(\bar{\varepsilon})$ of all the elements
 295 of the sample. Note that loading paths described as $\langle T_X \rangle = f(\bar{\varepsilon})$ are slightly
 296 different from those described by $T_X = f(\bar{\varepsilon})$. Figure 10 shows this envelope for
 297 the different sample geometries.

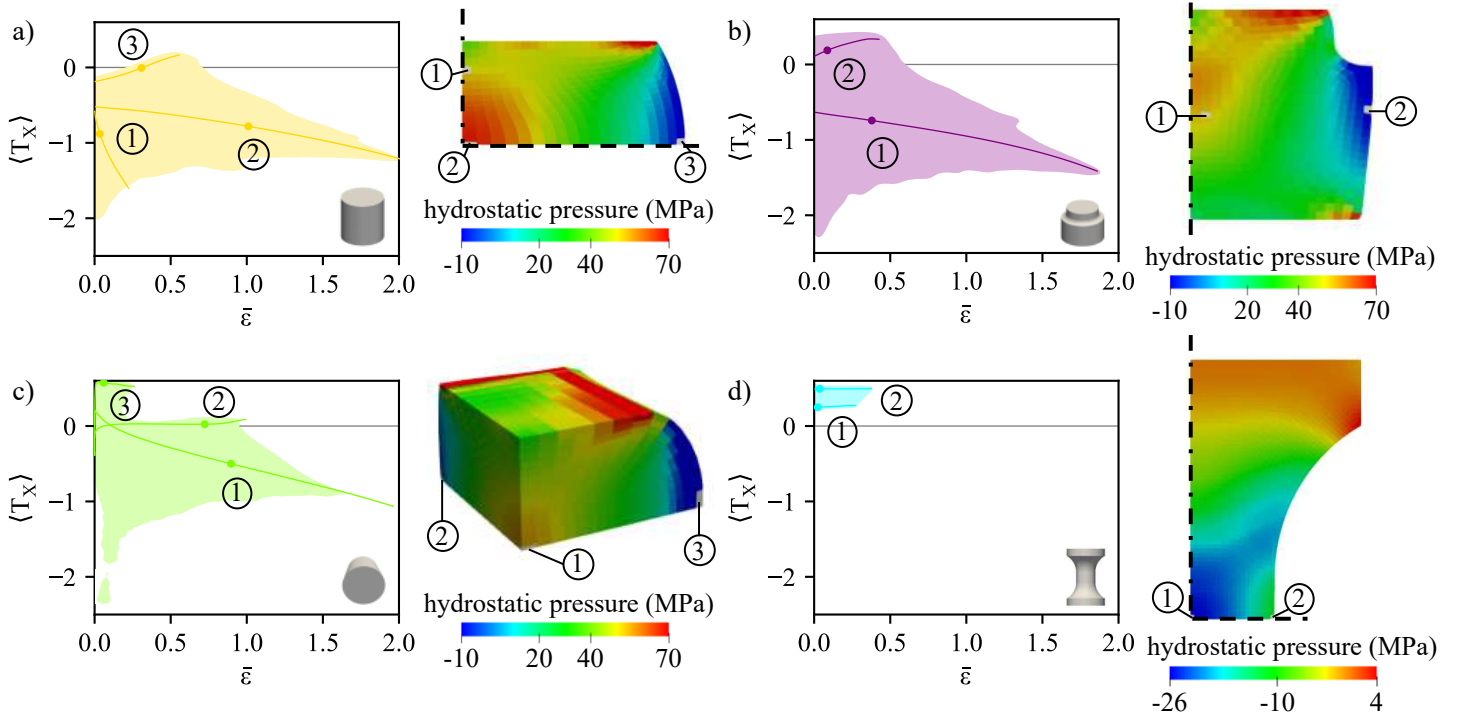


Figure 10: Loading domains and local triaxiality for the uniaxial tests: a) compression of a cylinder, b) compression of a double-collar, c) Brazilian compression of a cylinder, d) tension of a notched sample.

298 On the same Figure, local loading paths of some chosen elements are repre-

299 sented by lines. As for rolling, the loading path depends on the location within
300 the sample. During the compression of a cylinder (Figure 10.a), higher cumu-
301 lated strains are reached at the center of the sample with negative triaxiality (in
302 element 2). Triaxiality reaches positive values close to the border (in element 3)
303 after a local cumulated strain of 0.5. Tensile stresses appear due to the barrel
304 shape induced by friction between the dies and the sample. Close to the dies (in
305 element 1), strain is low. During the compression of a cylinder along its axis, a
306 very limited volume endures loading states with triaxiality ratios larger than or
307 even close to 0. Simulations with different alloys, temperatures and compression
308 speeds have not shown significant effects on loading paths.

309 Figure 10.b presents the loading domain for Brazilian tests. Large strains
310 at triaxiality close to zero are accessible in the elements at the center of the
311 cylinder base (in element 2). Tension due to the barrel effect is accentuated
312 during the test. High triaxiality ratios are reached, like in element 3, but strain
313 remains low.

314 Results for the simulations for double-collar samples are given in Figure 10.c.
315 For double-collar geometries, the upper part of the sample acts as an indent for
316 lower part. This results in positive triaxiality ratios even at early stage of
317 deformation.

318 Finally, with the tensile test presented on Figure 10.d positive triaxiality
319 ratios of about 0.33 can be reached with a low dispersion on loading paths.
320 With this geometry, there is no need to consider elements at the border of the
321 specimen to access positive stress triaxiality ratios.

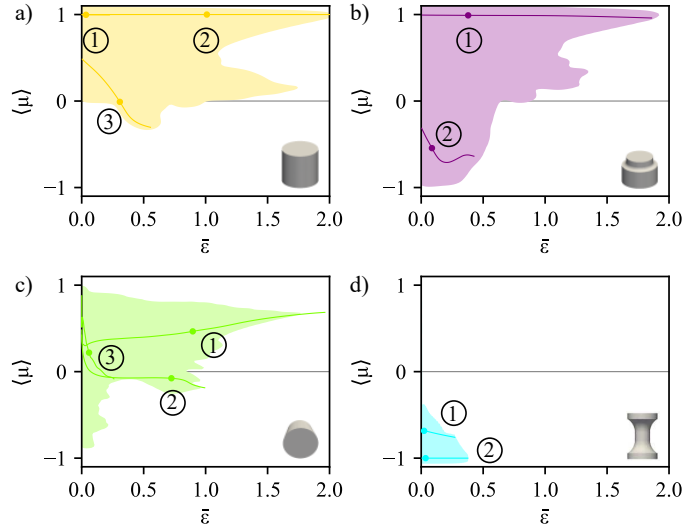


Figure 11: Loading domains and local Lode parameter for the uniaxial tests: a) compression of a cylinder, b) compression of a double-collar, c) Brazilian compression of a cylinder, d) tension of a notched sample. The local paths are those of elements presented on Figure 10.

322 Figure 11 presents the evolution of the Lode parameter during uniaxial tests.
 323 For deformation in compression (Figure 11.a-c) the value of the Lode parameter
 324 is mostly between $\langle \mu \rangle = 0$ (representative of plane strain) and $\langle \mu \rangle = 1$ (representative of uniaxial stress).
 325 With the compression of a cylinder or a double-collar,
 326 it is possible to reach negative values for the Lode parameter, in the elements
 327 close to the border of the sample. For the tensile test the Lode parameter is
 328 close to -1 which is representative of uniaxial tension.

329

330 In conclusion, the three loading zones of rolling previously described can be
 331 reproduced with uniaxial tests:

- 332 • At the edge of the plate (edge and extreme edge positions) and the center
 333 of the plate at low deformation $\langle T_X \rangle \geq 0$ and $\langle \mu \rangle \leq 0$. Both ranges of the
 334 Lode parameter and triaxiality are reproduced by the tensile test and also
 335 in zones close to the edges in compression of the cylinder, the double-collar
 336 and the Brazilian test.

- 337 • At the center of the plate at high deformation and the quarter thickness
338 $0 > \langle T_X \rangle \geq -0.7$ and $\langle \mu \rangle = 0$. The range of triaxiality is reproduced
339 by the different types of compression tests. There is a variability of the
340 Lode parameter between 1 and 0 in those samples. Chbihi et al. (2017)
341 found that for a given pore at $\langle T_X \rangle = -0.33$, the closure strain is 0.6 at
342 $\langle \mu \rangle = 1$ and it is 0.75 for $\langle \mu \rangle = 0$. On the other hand, Saby et al. (2015a)
343 observed that the closure strain varies by twice on ellipsoids when different
344 orientations or shape factors are considered (for $\langle T_X \rangle = -0.33$). In the
345 present case of real casting pores with various orientations and shapes, the
346 effect of the Lode parameter in this range of triaxiality will be of second
347 order compared to the effect of morphology. For this zone we will only
348 focus on the reproduction of triaxiality.
- 349 • At the surface $\langle T_X \rangle < -0.7$ and $\langle \mu \rangle = 0$. The triaxiality is highly nega-
350 tive, the closure is controlled by the value of the triaxiality and the Lode
351 parameter has a negligible effect. Such loading conditions are fulfilled at
352 the center of the sample under uniaxial compression.

353 The combination of the different geometries of uniaxial tests enables to reach
354 all the final loading states of rolling. Note that this study gives all the accessible
355 loading states in in situ samples, however, experimentally pores are not present
356 in every element of every sample. Thus, it is necessary to perform several tests
357 with the same sample geometry to increase the chance to have pores in regions
358 of interest.

359

360 3.3. Example of experimental results

361 Figure 12 shows the experimental results for a double-collar sample, with the
362 3D evolution of pores within the sample (Figure 12.a). The relative reduction
363 (r) is computed with the initial and current height of the sample (it is not linked
364 to that of rolling). The strain is inhomogeneous in the sample, the upper part of
365 the sample with a smaller radius deforms more and deformation is progressively

366 transmitted to the lower part. In consequence, pores start to close in the upper
367 part of the sample and closure propagates to the lower part close to the axis
368 of symmetry whereas pores close to the border evolve less. Sample geometry
369 used for simulation is shown on Figure 12.b. It is generated from measurement
370 on tomographic volume and thus differs from geometries previously simulated.
371 We can observe several tens of pores in the sample. Figure 12.c shows the load-
372 ing paths of two clusters of pores together with their morphological evolution.
373 Loading path is obtained with simulation and morphological evolution thanks
374 to DVC. Cluster A which experiences a triaxiality close to zero has few changes
375 in shape whereas cluster B that is subjected to higher negative triaxiality and
376 higher cumulated strain progressively closes.

377 Such results are accessible for all the pores in all the tested samples. Thus,
378 closure kinetics of real pores with large ranges of triaxiality and cumulated
379 strain, comparable to those of hot rolling, can be studied.

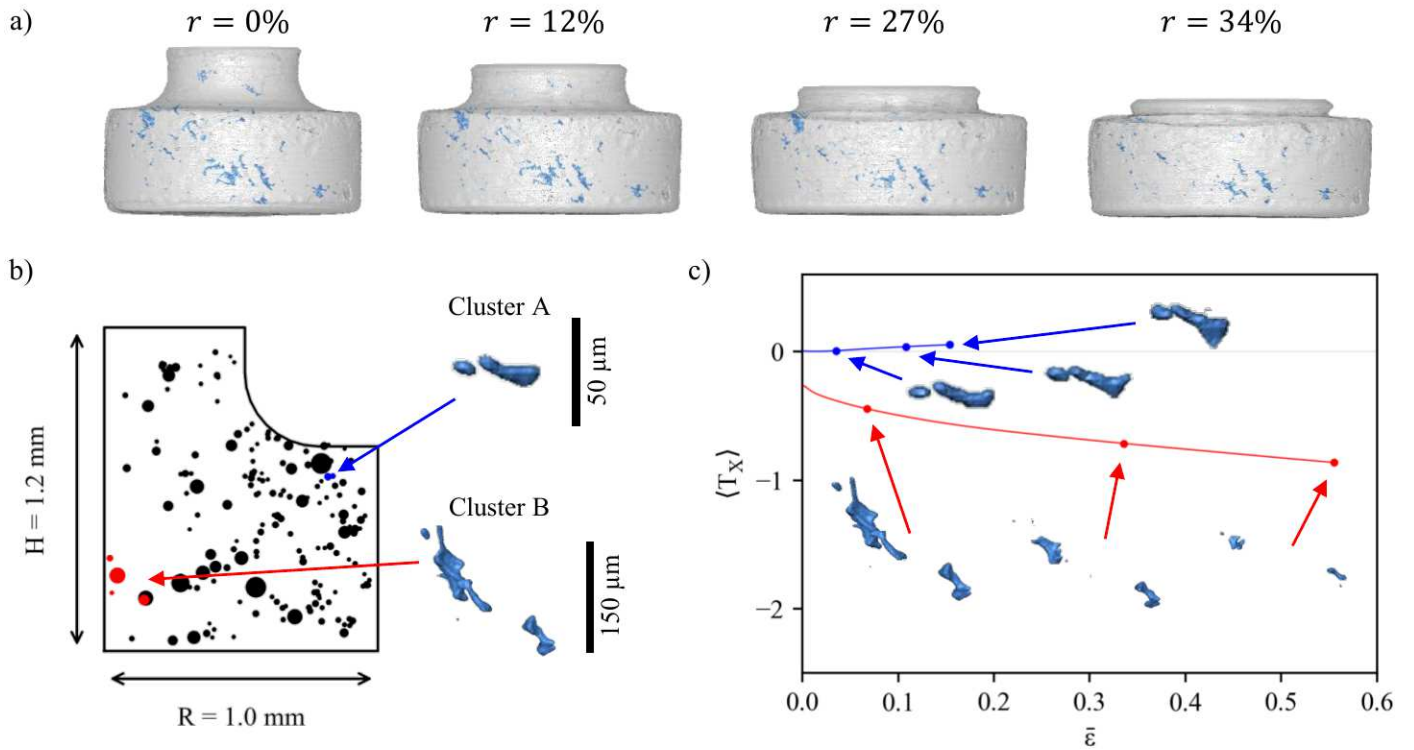


Figure 12: Experimental results of the volume evolution of pores in a double-collar sample. a) 3D rendering of the pores (in blue) in the sample during compression. b) Initial state: schematic representation of all the pores (pores are represented by black points with a diameter equal to the pore equivalent diameter, they are projected in the axisymmetric geometry used for simulation) and 3D rendering of two clusters of pores. c) Loading path and morphological evolution of the two considered clusters.

380 *3.4. On the choice of loading paths descriptors*

381 The hydrostatic integration (noted Q in Equation 8) is often used to describe
 382 pore closure, as reviewed by (Saby et al., 2015b). It is equal to the product of
 383 the two loading state descriptors chosen before, namely the average triaxiality
 384 and the cumulated strain. The hydrostatic integration takes into account the
 385 variation of triaxiality during the test and it is computed locally to account for
 386 the inhomogeneities of mechanical fields in the sample.

$$Q(\bar{\varepsilon}) = \int_0^{\bar{\varepsilon}} T_X(\bar{\varepsilon}) d\bar{\varepsilon} = \langle T_X \rangle \bar{\varepsilon} \quad (8)$$

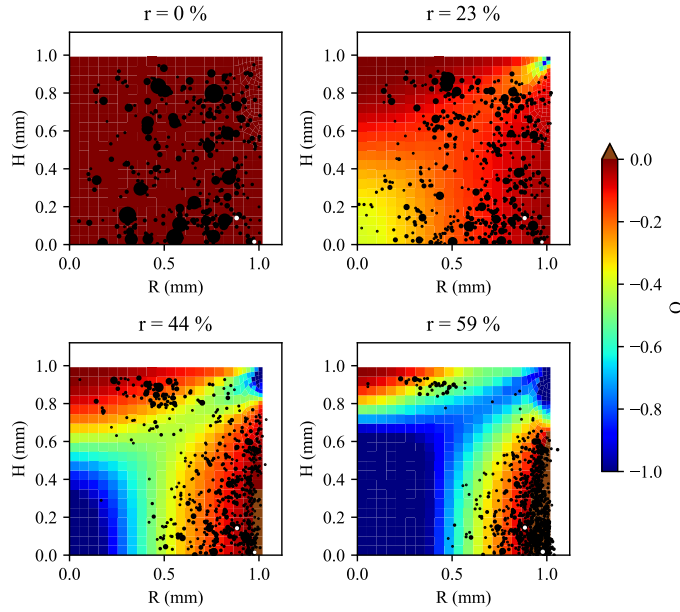


Figure 13: Superposition of the hydrostatic integration field Q and the pores present in a cylindrical sample during its compression. Pores are represented by black points with a diameter equal to the pore equivalent diameter. Everything is represented in the initial state geometry and in the axisymmetric plane, taking into account the axial and planar symmetries of the cylinder. In white, tracking of two pores in zone of low $|Q|$.

387 Figure 13 shows the qualitative correlation between the hydrostatic integra-
 388 tion and the evolution of the volume of real pores. The colored background rep-

389 represents the hydrostatic integration field. Pores are represented by black points
390 of equivalent diameters (meaning that the point diameter is equal to the pore
391 equivalent diameter). Hydrostatic integration field and pores are represented in
392 the initial configuration. Along deformation, hydrostatic integration decreases
393 in most of the sample. On the edge, positive values progressively appear, be-
394 tween $r = 23\%$ and $r = 44\%$, due to the barrel shape induced by the friction
395 between the dies and the sample. Close to the surface in contact with the die,
396 hydrostatic integration remains low due to low strain. Conversely, in the upper
397 right corner Q decreases with average strain because the lateral surface of the
398 cylinder progressively comes into contact with the die leading to large strains.

399 Hydrostatic integration variations correlate well with pore volume varia-
400 tions. When Q is positive, pores grow and new pores nucleate whereas they
401 shrink where Q is negative and shrinkage is more pronounced where the abso-
402 lute value of Q is high. Contrary to what Nakasaki et al. (2006) and Kakimoto
403 et al. (2010) stated, pores are still present when $Q < -0.2$ and even for lower
404 values. Here, pores are totally closed for values around $Q = -1$.

405

406 The good qualitative correlation between the hydrostatic integration and the
407 pore shrinkage or growth shows that mean triaxiality and cumulated strain are
408 relevant descriptors to focus on in order to reproduce loading paths.

409 4. Conclusions

410 In situ mechanical tests characterized by X-ray microtomography can be
411 used to acquire experimental data on real pore volume evolution under defor-
412 mation representative of hot rolling. Nevertheless, this method requires small
413 samples (with dimensions of a few millimeters) whereas industrial rolling in-
414 volves plates at the meter scale. To ensure that the loading states accessible
415 with uniaxial tests are representative of the loading states of rolling, FE sim-
416 ulations of both industrial rolling at the meter scale and uniaxial tests at the
417 millimeter scale were performed. The loading states that can be experienced

418 by pores in terms of triaxiality and cumulated strain were compared. Based on
419 these simulations, the following conclusions can be drawn:

- 420 • When a single pass of rolling is considered, the use of the mean triaxiality
421 during deformation and the final cumulated strain gives a good approxi-
422 mation of the local loading state experienced during the pass. Their values
423 depend on the considered position in the plate. The relative reduction of
424 the pass has a direct influence on the cumulated strain and the triaxiality
425 value is linked to the L/H ratio.
- 426 • The combination of several sample geometries deformed uniaxially allows
427 all the loading states experienced during thick plate hot rolling to be
428 reproduced. Selected tests are: compression of a cylinder along its axis,
429 Brazilian test, double-collar compression and tensile test on a notched
430 sample. In cases where the Lode parameter affects pore volume evolution,
431 for low absolute values of triaxiality, those geometries also reproduce the
432 ranges of values of the Lode parameters encountered during rolling.
- 433 • A dedicated sample environment integrated within a synchrotron beamline
434 was developed to capture samples evolution during interrupted in situ tests
435 at high temperature with X-ray microtomography. An automated data
436 processing workflow combining images processing, DVC and FE simulation
437 was set up. It allows the morphological evolution of each pore to be linked
438 to the local mechanical fields it experiences during the deformation.
- 439 • The average triaxiality $\langle T_X \rangle$ and cumulated strain $\bar{\varepsilon}$ have been used to
440 compare loading paths encountered during rolling and during uniaxial
441 tests. This choice is justified by a qualitative comparison of the hy-
442 drostatic integration field, combining both parameters, and the locations
443 where pores shrink or grow.
- 444 • From the volume evolution of pores with the hydrostatic integration, it
445 can be deduced that pores closure is enhanced by high cumulated strain

446 and high negative triaxiality. They can be reached during rolling with a
447 high relative reduction and high values of L/H ratio.

448 Thus, with the method described in this article, experimental data on the
449 evolution of real pores are acquired during loading representative of hot rolling.
450 For the sake of clarity, the conclusions on pore closure are limited in this paper.
451 Nevertheless, this method can have many applications. It could be used, on
452 the one hand, to obtain a better description of pore closure or opening kinetics
453 by studying pore volume evolution as a function of the hydrostatic integration.
454 It also gives access to fine morphological evolution in 3D during deformation
455 which will allow mechanisms involved in pore closure or opening to be studied.
456 Finally, the database of hundreds of pores could be used to test the different
457 models of pore volume evolution available in the literature.

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