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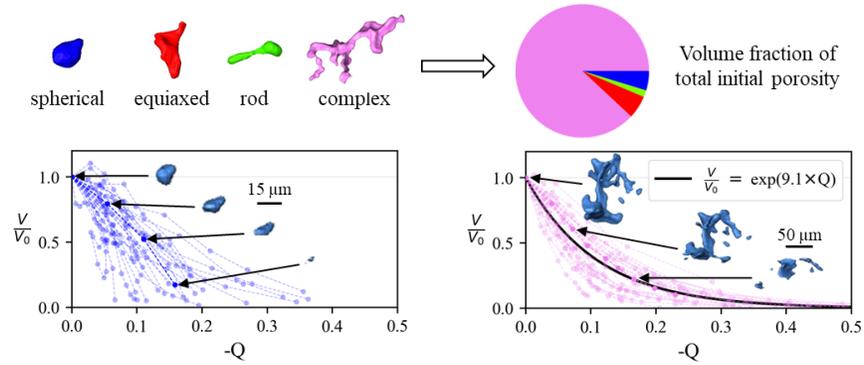
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Graphical Abstract

Mechanisms and kinetics of pore closure in thick aluminum plate

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Mechanisms and kinetics of pore closure in thick aluminum plate

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Abstract

Interrupted in situ tests at high temperature characterized with X-ray microtomography were employed to study porosity evolution under loading conditions representative of aluminum hot rolling. Coupling digital volume correlation and simulations, it is possible to access, for each pore, its morphological evolution, and the mechanical fields it experienced. The closure kinetics of hundreds of pores were studied, which is described as the pore volume as a function of the hydrostatic integration. Closure kinetics are a function of the shape of the pore. Focus is put on pores with a complex shape which are more detrimental to mechanical properties. Their closure kinetics are exponential due to the pore fragmentation during closure. The closure of complex pores is fostered by an initial orientation perpendicular to the loading direction and a low initial fragmentation. Based on the closure kinetics of 243 complex pores, a new pore closure model is proposed.

Keywords: Aluminum, Porosity, Closure kinetics, In situ X-ray microtomography, Pore closure model

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1 **1. Introduction**

2 Thick aluminum plate is widely used in aerospace applications to machine
3 structural aircraft elements. During casting, porosity appears in the ingot due
4 to the higher density of solid aluminum and the lower hydrogen solubility in
5 solid aluminum compared to liquid aluminum. Pores must be closed during the
6 transformation because their presence in the final product would significantly
7 decrease the mechanical properties of the material. The fatigue life of the mate-
8 rial is reduced (Couper et al., 1990) as well as the ductility and tensile strength
9 (Wu et al., 2012). Hot rolling is the processing step which enables to close the
10 pores in these thick plates.

11
12 Park and Yang (1996) demonstrated that pore closure occurs in two steps.
13 First morphology and topology of the pore evolve, along with the pore volume
14 decrease. Then, the internal surfaces of the pore weld together during bound-
15 ing. In the following, we will only focus on the first step also called mechanical
16 closure by Saby et al. (2015b). The present work aims at a better understanding
17 of pore closure mechanisms and at providing a better description of pore volume
18 evolution during pore closure.

19
20 From the literature, we can report that mechanical fields expressed through
21 the triaxiality and cumulated strain play a major role in pore closure, they
22 were used by Gurson (1977), Duva and Hutchinson (1984), Zhang et al. (2009)
23 and Saby et al. (2015a). Besides, closure also depends on pore shape and ori-
24 entation, as shown by Saby et al. (2015a) who developed a closure model for
25 ellipsoidal pores taking into account the orientation and the shape of the pore.
26 Llanos et al. (2008) observed that natural holes are harder to close than artificial
27 drilled holes. Nevertheless, most of the experiments and numerical simulations
28 are conducted on simple pore geometries (spherical or ellipsoidal) whereas real
29 casting pores can be branched and tortuous. The present paper investigates,
30 using in situ X-ray microtomography, the closure of real pores experiencing

31 loading paths representative of hot rolling.

32

33 **2. Experimental methods**

34 The experimental methods are detailed in Gravier et al. (under revision
35 - PROTEC-D-21-02245). In summary, in situ mechanical tests at high tem-
36 perature characterized by X-ray microtomography are performed to mimic hot
37 rolling loading and observe porosity evolution during deformation. Tests are
38 performed in a synchrotron (ESRF-ID19, Grenoble, France), providing volumes
39 with a voxel size of $2.44 \mu m$ where the whole sample and pores are visible.

40 This method provides 3D images of samples. Post treatment based on im-
41 age processing, Digital Volume Correlation (DVC) and simulations allows the
42 morphological evolution of tens of pores in each sample to be obtained as well
43 as the local loading paths they experienced.

44 For the following study of pore closure, we will use data provided by the
45 compression of cylindrical samples. Their dimensions are about 2 mm in diam-
46 eter and 2 mm in height, they are made of 2XXX series alloy in which porosity
47 was purposely increased. The samples are extracted in casting ingot after ho-
48 mogenization. The microstructure is typically made of equiaxed grains with an
49 average grain size of $180 \mu m$ and a random texture. In these samples, the vol-
50 ume fraction of porosity ranges between 2.5×10^{-4} and 8.5×10^{-4} . The volume
51 fraction of intermetallics is around 3×10^{-3} . For the compression, loading is ap-
52 plied along the height of the samples (which will be the vertical direction on the
53 following figures). Five samples are compressed at $480 \text{ }^\circ\text{C}$ with a compression
54 speed of $1000 \mu m.s^{-1}$, we will refer to these conditions as standard conditions.
55 Two different temperatures (455 and 520°C) and two lower compression speeds
56 (100 and $500 \mu m.s^{-1}$) are also tested.

57 **3. Porosity description**

58 Each sample typically contains tens of pores (Figure 1.a). However, be-
59 cause of the resolution of tomography, the precise number of pores is difficult
60 to determine unambiguously, as apparently separate pores may in reality be
61 connected by unresolved thin ramifications. Also, some of the pores are close
62 to each other and may interact during deformation (Figure 1.b). In order to
63 describe precisely the tomographic observations, we will use here the concepts
64 of clusters and subpores. Subpores are all the pores that are separate from
65 one another after thresholding (Figure 1.c). When these subpores are less than
66 50 μm apart (or 20 voxel surface to surface distance), they belong to the same
67 cluster (Figure 1.d). The distance of 50 μm was chosen such that big clusters do
68 not merge. It should be noted that the standardized dye penetrant inspection,
69 used to validate industrial plate production, quantifies objects similar to these
70 clusters: Kishimoto et al. (1989) demonstrated that if pores are close to each
71 other, they can be considered as the same pore. The porosity is composed by
72 the sum of all the clusters contained inside the sample. If we are able to predict
73 the volume evolution of each single cluster, we are then able to compute the
74 volume evolution of porosity.

75 A cluster can be composed of one or more subpores. A cluster's fragmen-
76 tation is defined as the number of subpores it contains. As the tomographic
77 resolution might be too coarse to image thin ramifications between subpores,
78 fragmentation tends to be overestimated. Nevertheless, subpores that appear
79 separate due to the resolution limitations but are actually connected will be
80 contained within the same cluster. Thus, in this paper the volume evolution of
81 pores will be investigated at the scale of clusters.

82

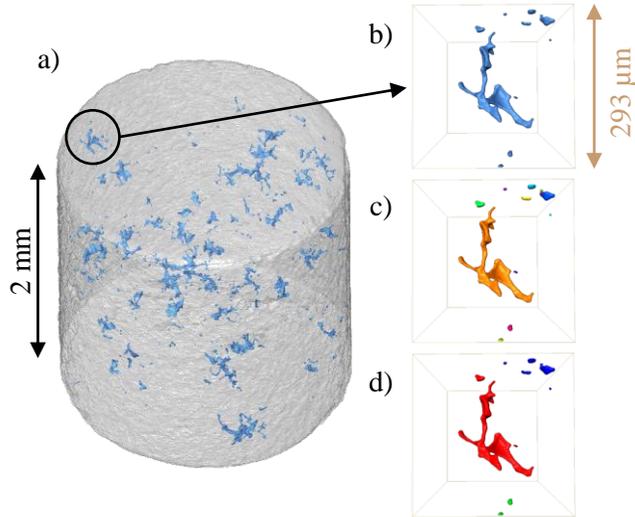


Figure 1: a) 3D rendering of pores (in blue) within a cylindrical sample. b) Zoom on pores. c) Pores colored by subpores: each individual pore has a different color. d) Pores colored by clusters: pores less than 50μm apart belong to the same cluster and have the same color.

83 The pores present in the samples have various shapes. They appear at
 84 solidification due to two concurrent mechanisms as reported by Dantzig and
 85 Rappaz (2016). On the one hand, the lack of mass feeding to compensate for
 86 the volumetric shrinkage causes the formation of pores in the interdendritic
 87 region. These pores are branched and tortuous. On the other hand, the lower
 88 hydrogen solubility in solid aluminum compared to liquid aluminum results in
 89 the liquid enrichment in gas. The gas is rejected under the shape of bubbles
 90 forming spherical pores. Shape has been reported to have a strong influence
 91 on pore closure. Llanos et al. (2008) observed differences between artificial and
 92 drilled pores, Chen et al. (2010) reported differences in the simulated closure
 93 of tetrahedral and spherical pores and Saby et al. (2015a) took into account

94 shape factors in the closure of ellipsoids. Based on these observations, pores are
 95 classified into 5 categories: spherical, equiaxed, rod, sheet and complex. The
 96 shape classification is adapted from the work of Fang et al. (2016) and is applied
 97 to subpores. Shapes are determined as a function of three parameters (Tab. 1):
 98 pore elongation E (Eq. 1), flatness F (Eq. 2) and complexity C (Eq. 3).

$$E = \frac{2a}{b+c} \quad (1)$$

$$F = \frac{b}{c} \quad (2)$$

99 Where $a > b > c$ are the semi-axes of the pore equivalent ellipsoid, namely
 100 the ellipsoid with the same inertia matrix (see Figure 2).

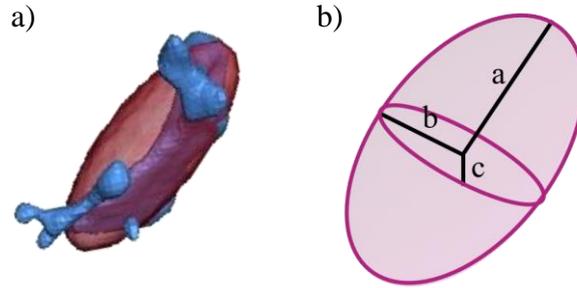


Figure 2: a) 3D rendering of a pore (in blue) with its equivalent ellipsoid (in red) that have the same inertia matrix as the pore. b) Semi-axes of the equivalent ellipsoid, $a > b > c$.

$$C = \frac{1}{s} = \frac{1}{6V} \sqrt{\frac{A^3}{\pi}} \quad (3)$$

101 s is the sphericity defined as the ratio of the pore volume V over the volume
 102 of a sphere having the same surface area A . For a sphere $s = 1$, $s < 1$ otherwise.

Shape	E	F	C	Color code
Spherical	/	/	$C \leq 1.5$	blue
Equiaxed	< 4	< 5	$1.5 < C < 2.5$	red
Rod	≥ 4	< 5	$1.5 < C < 2.5$	green
Sheet	/	≥ 5	$1.5 < C < 2.5$	yellow
Complex	/	/	$C \geq 2.5$	pink

Table 1: Shape classification

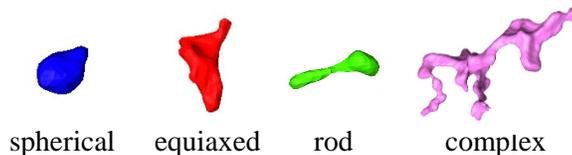


Figure 3: Examples of 3D views of different pore shapes according to the classification defined in table 1. Note that according to this classification, spherical pores can have protrusions and equiaxed pores can have small branches.

103 Examples of subpores of each category are shown in Figure 3, no flat subpores
104 corresponding to sheet category were found in the present studied samples.
105 Color code defined in table 1 will be used in all figures when shape classification
106 is involved. In the following, simple shapes will refer to shapes with a low
107 complexity, namely spherical, equiaxed and rod shapes.

108 A shape is also attributed to each cluster. Given that a cluster can be
109 composed of subpores with different shapes, the cluster shape is the shape which
110 is the most represented in volume fraction.

The size of a subpore (or a cluster) will be described with the equivalent diameter D_{eq} which is the diameter of a sphere having the same volume V as the subpore (or cluster) (Equation 4).

$$D_{eq} = 2 \left(\frac{3}{4\pi} V \right)^{\frac{1}{3}} \quad (4)$$

111 Finally, orientation is defined at the cluster scale using equivalent ellipsoid.
 112 The semi-axes of the ellipsoid ($r_1 > r_2 > r_3$) and their respective direction
 113 ($\vec{u}_1, \vec{u}_2, \vec{u}_3$) are computed from the inertia matrix of the cluster. Cluster orienta-
 114 tion is the value of the angle between the direction of the major axis (\vec{u}_1) and
 115 the direction of macroscopic loading (vertical direction in the following figures).
 116

117 4. Results and discussion

118 4.1. Shape and size of subpores in the initial state

119 Figure 4 shows the distribution of subpores as a function of their shape and
 120 their equivalent diameter. These are subpores present in the initial state in a
 121 given sample.

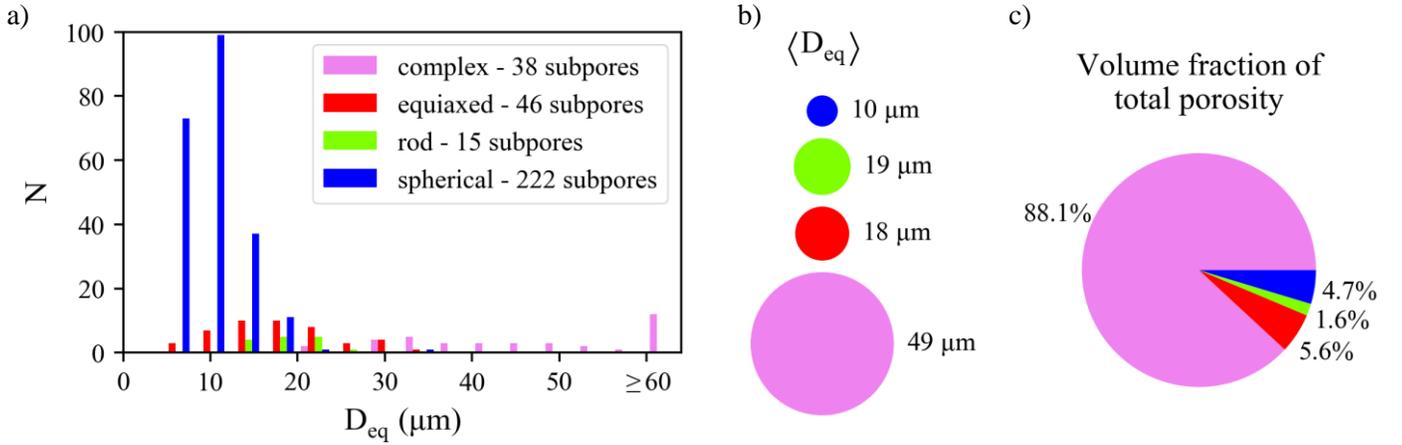


Figure 4: Shape and size of subpores in the initial state for a given sample. a) Distribution of subpores as a function of their equivalent diameter and shape. b) Mean equivalent diameter of subpores per shape. c) Contribution of each shape on the total volume of porosity.

122 The biggest subpores are complex with an equivalent diameter globally over
 123 $20 \mu m$ (Figure 4.a), their average equivalent diameter is $49 \mu m$ (Figure 4.b).

124 Conversely, spherical subpores have a low equivalent diameter, generally under
125 $20 \mu m$ (Figure 4.a). Note that no quantification of subpores was performed
126 below a volume of 8 voxels to avoid artifacts due to image noise. At such a
127 small volume (equivalent diameter of $6 \mu m$), the distribution of voxels cannot
128 form a complex shape, so small pores are more likely to appear spherical due to
129 resolution limits. Equiaxed and rod subpores have intermediate sizes between
130 those of spherical and complex subpores.

131 Even if spherical subpores are more numerous, complex subpores represent
132 88% of the total volume of porosity (Figure 4.c). Complex subpores represent
133 most of the porosity. Furthermore, they are the biggest, making them poten-
134 tially more harmful for mechanical properties. Couper et al. (1990) demon-
135 strated that the fatigue life of the material is increased when pores are smaller.
136 Based on these observations, the description of closure of complex clusters is a
137 key issue.

138 *4.2. Shape evolution of subpores during closure*

139 During the compression of cylindrical samples, porosity decreases because of
140 negative triaxiality in most of the volume and large plastic deformation. Due to
141 friction with the dies, the deformation is not homogeneous in the sample: zones
142 close to the dies hardly deform and the sample takes a barrel shape. It induces
143 tensile stresses on the sides of the samples which prevent pore closure and even
144 lead to pore growth or pore nucleation. Therefore, at the end of the compression,
145 pores are closed at the center of the sample and some pores may remain on the
146 edges or may even appear in the last step of compression. Figure 5 shows the
147 3D rendering of pores within a cylindrical sample during compression. Most of
148 the pores initially present are closed after a reduction of 59 %. Few pores are
149 still present at the end of the test: on the edges due to tension and close to the
150 top and bottom surface due to low deformation in these regions.

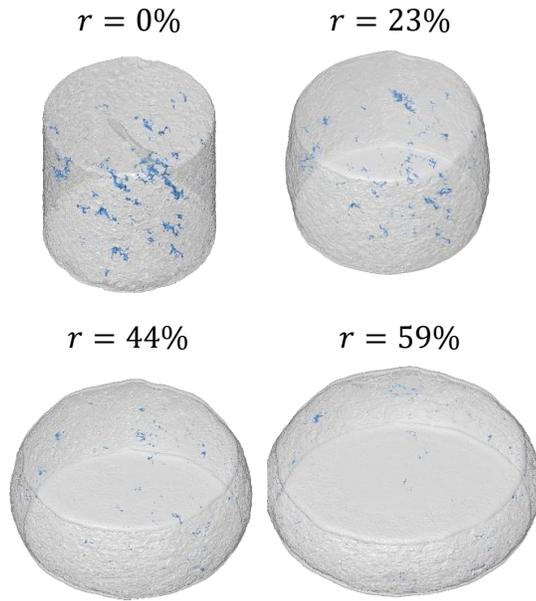


Figure 5: 3D rendering of pores (in blue) within a cylindrical sample during its compression.

151 At every compression step, a shape was assigned to each subpore. This
152 shape is independent of the subpore history meaning that a subpore can have
153 different shapes at different deformation steps. For each shape category, average
154 equivalent diameter and number of subpores are plotted in Figures 6.a. and b.

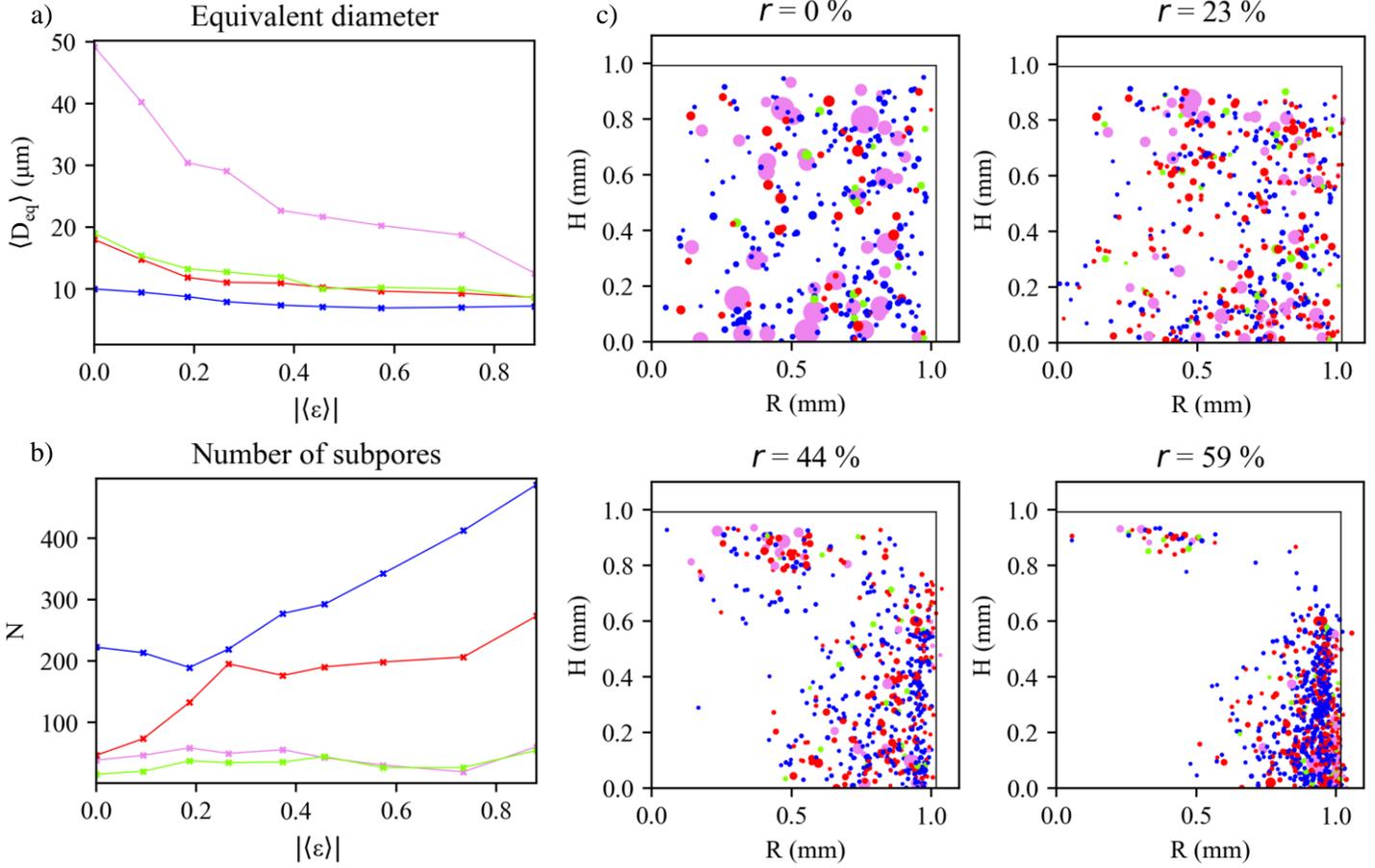


Figure 6: Evolution in size and number of subpores during compression depending on their shape. a) Evolution of the average equivalent diameter per shape. b) Evolution of the number of subpores per shape. c) Subpores position, size and shape in the axisymmetric projection. Position is represented in the initial state using the estimated initial position given by DVC. The deformation of the sample is not represented to ease the visualization of tracking.

156 decrease due to total closure or coalescence. The number of complex subpores
157 remains constant during the early stage of deformation but their average equiv-
158 alent diameter decreases sharply. This can be explained by the fragmentation
159 of complex pores into a smaller complex pore and other simpler shapes such
160 as equiaxed pores. The increase in the number of equiaxed pores supports this
161 argument. On the other hand, the number of spherical pores decreases at the
162 beginning of compression meaning that there is more spherical pores that close
163 than spherical pores that appear due to fragmentation. After a strain of 0.3, the
164 number of equiaxed pores reaches a plateau: there is an equilibrium between
165 the creation of equiaxed pores from the fragmentation of bigger complex pores
166 and the fragmentation of equiaxed pores into spherical pores.

167 This fragmentation of large complex subpores to smaller subpores of simpler
168 shapes is visible on Figure 6.c. Subpores are projected on the axisymmetric
169 plane. For each deformation step, subpore position is represented in the initial
170 state using the estimated initial position given by DVC. Thus, deformation of
171 the sample is not represented. Using initial configuration representation helps
172 subpore tracking: if subpores are located at the same position at different defor-
173 mation steps, they are likely to be the same subpore. Subpores are represented
174 by disks of equivalent diameter (meaning that the disk diameter is equal to
175 the subpore equivalent diameter). Disk color describes the associated subpore
176 shape. Complex subpores decrease in size and split into new equiaxed, rod or
177 spherical subpores. Equiaxed subpores become spherical and finally close. This
178 Figure also shows the nucleation of small spherical subpores close to the edges.
179 It is correlated with the final increase in number of spherical subpores. Finally,
180 subpores located near the dies do not close because of low strain in this zone.

181

182 *4.3. Morphology evolution of clusters during closure*

183 The evolution of individual cluster morphology has been characterized by the
184 cluster fragmentation and the shape evolution of the subpores in the cluster.

185 For spherical clusters, there is no fragmentation. Cluster are generally com-

186 posed of a single subpore. The subpore closes progressively, remaining spherical
187 or sometimes becoming equiaxed due to flattening. The final description of
188 spherical pore closure is limited by the resolution of tomography. In contrast,
189 complex clusters are initially composed of several subpores. They are generally
190 made of a central big complex subpore surrounded by smaller subpores. It is
191 possible that these subpores actually connect but that the resolution limit does
192 not enable to see the connection. An example is given on Figure 7. In the
193 present case, the cluster is initially composed of 2 complex subpores with the
194 biggest at the center. They are surrounded by 8 smaller subpores (rod, equiaxed
195 or spherical). The figure describes the evolution of the composition of the cluster
196 during the test. After the first compression step, the smallest complex subpore
197 (at the upper left) fragments into a rod and a spherical subpores. The main
198 complex subpore fragments also but generates only small subpores with simple
199 shapes (from scan 1 to 3). Most of the volume remains in the complex subpore
200 until scan 4. From then on, the big complex subpore fragments into 4 smaller
201 complex subpores of similar volumes and other simple subpores. From scan 6,
202 these complex subpores evolve to simpler shapes and finally close. The overall
203 number of subpores first increases, owing to fragmentation and then decreases
204 due to pore closure.

205 3D renderings also show that when the cluster has lost most of its volume,
206 its dimension is still large. At scan 4, only 24% of the pore volume remains
207 whereas its dimension perpendicular to the compression direction seems to re-
208 main unchanged. The cluster can still be detrimental for mechanical properties
209 even when it reaches a low relative volume because, as observed by Murakami
210 and Endo (1994), the pore has a crack shape. Therefore, it is necessary to
211 describe finely the final closure of clusters up to a very small remaining volume.

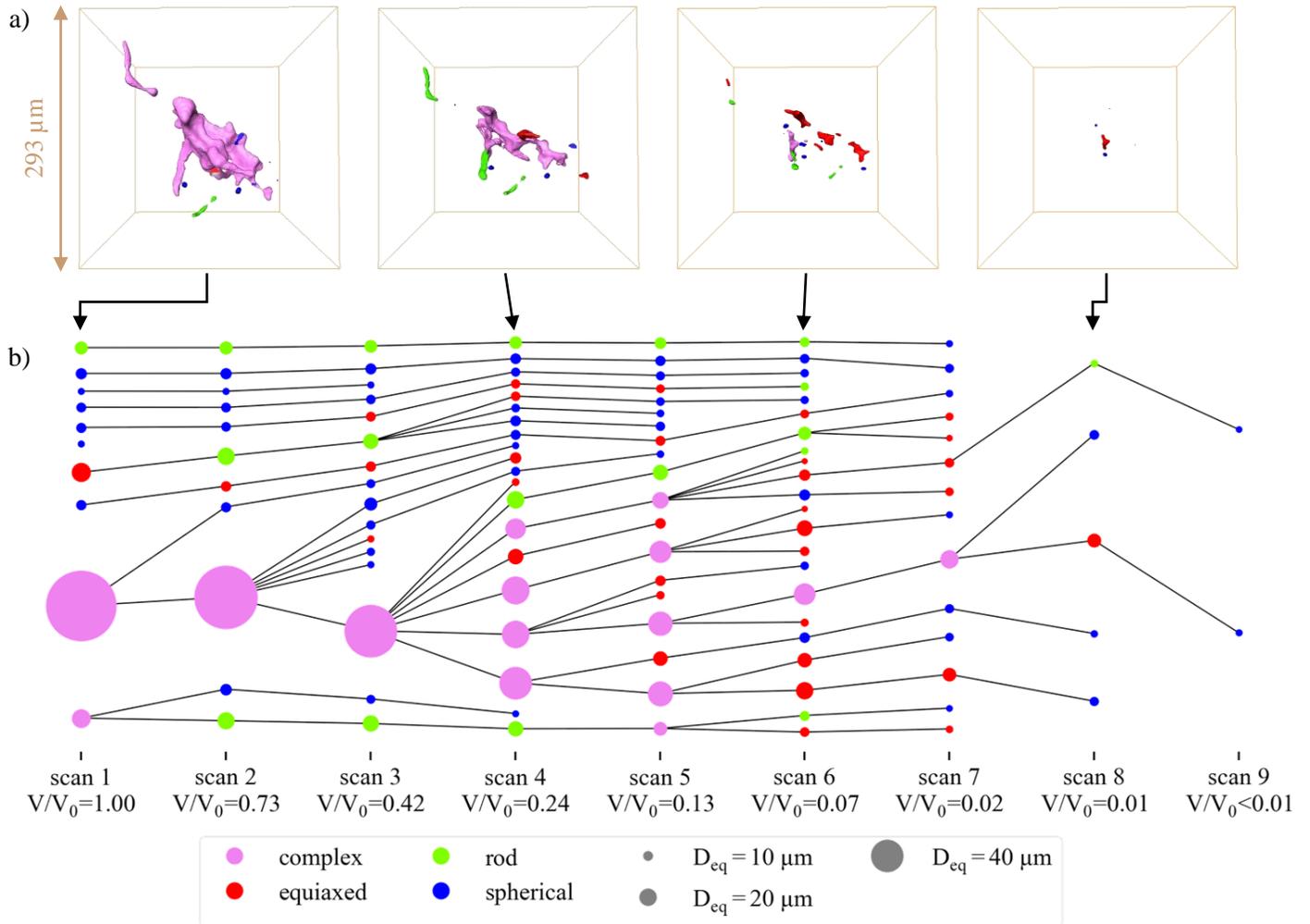


Figure 7: Fragmentation of a complex cluster during closure. a) 3D rendering of the cluster at different compression steps (loading was applied in the vertical direction). b) Schematic representation of the subpores composing the cluster.

212 4.4. Closure kinetics

213 According to literature, triaxiality T_X and cumulated strain $\bar{\epsilon}$ are the two
 214 main parameters controlling pore closure. The hydrostatic integration Q (Equa-

215 tion 5) proposed by Tanaka et al. (1987) combines these parameters. Qualita-
 216 tively, pore volume evolution correlates well with Q . Pore closure kinetics will
 217 be described by the evolution of the volume of pore clusters as a function of the
 218 hydrostatic integration Q .

$$Q(\bar{\varepsilon}) = \int_0^{\bar{\varepsilon}} T_X(\bar{\varepsilon}) d\bar{\varepsilon} = \langle T_X \rangle \bar{\varepsilon} \quad (5)$$

219 Recent pore closure models developed by Chbihi et al. (2017), Zhang et al.
 220 (2020), Wang and Dong (2020) take into account the Lode parameter μ in
 221 addition to the triaxiality. The influence of the Lode parameter affects the
 222 evolution of the pore volume when the triaxiality is close to zero. In the present
 223 study, $\langle T_X \rangle \leq -0.1$ and $0 \leq \mu \leq 1$. Wang and Dong (2020) found that for a
 224 given pore at $\langle T_X \rangle = -0.3$, the closure strain is 0.55 for $\langle \mu \rangle = 1$ and it is 0.75
 225 for $\langle \mu \rangle = 0$. On the other hand, Saby et al. (2015a) observed that the closure
 226 strain varies by twice on ellipsoids when different orientations or shape factors
 227 are considered. In the present case of real casting pores, the effect of the Lode
 228 parameter will be neglected compared to the effect of morphology. Thus we will
 229 not take the Lode parameter into account to study closure kinetics. The pores
 230 studied here experience stress states representative of the center of the plate
 231 or close to the rolls during rolling. They are not representative of the edges
 232 of the plate where the triaxiality is positive or close to zero. More details on
 233 the reproduction of stress states are given in Gravier et al. (under revision -
 234 PROTEC-D-21-02245).

235 Closure kinetics are studied observing clusters in compressive zones of cylindrical
 236 samples deformed by compression. Closure kinetics of all the clusters in a given
 237 cylindrical sample are presented on Figure 8.a. As hydrostatic integration is
 238 negative and decreases along deformation, $-Q$ is used for the X-axis. Only
 239 points with non-zero volume are represented on these graphs. When volume
 240 is equal to zero it is not possible to determine if clusters are really closed or if
 241 they are no longer detected (lost during tracking or volume lower than detection
 242 limit of 8 voxels). In the following, clusters with an initial volume of less than

243 40 voxels ($= 581 \mu m^3$) were not considered because their volume is lower than
 244 8 voxels when they reach 20% of their initial volume. For such small clusters,
 245 final closure cannot be studied.

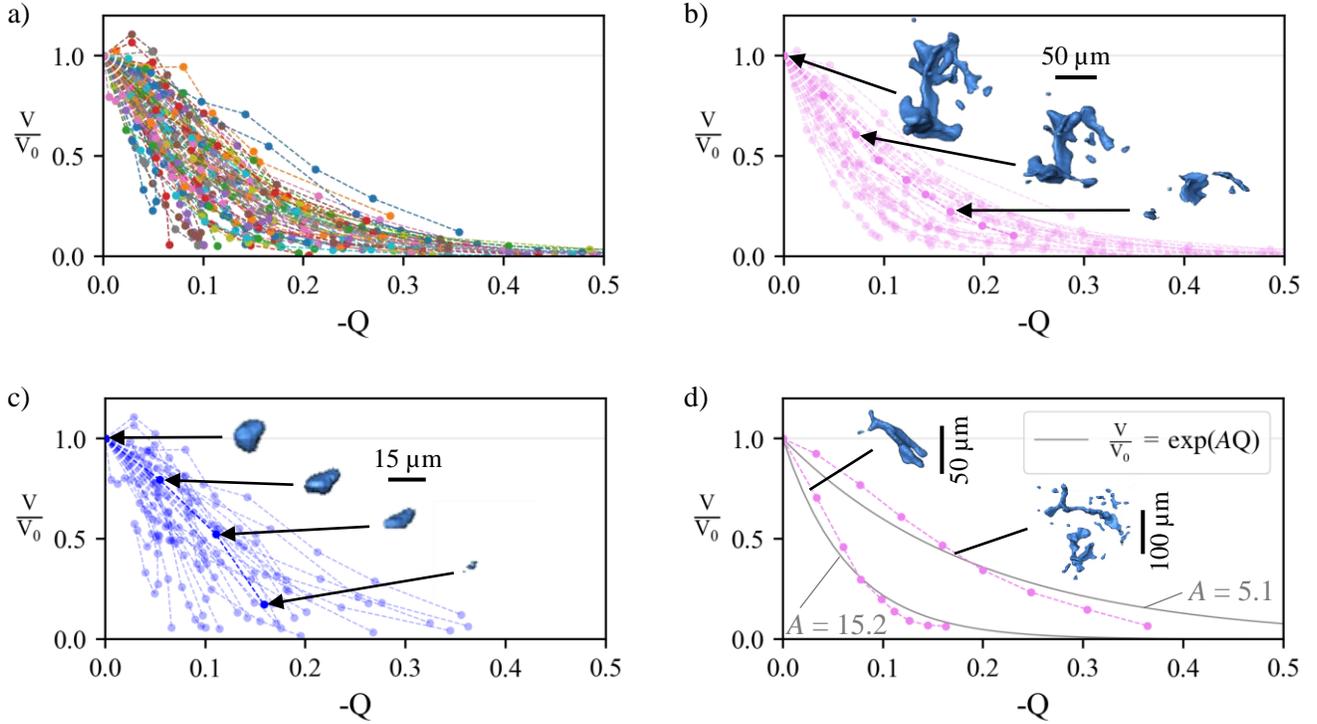


Figure 8: Cluster closure kinetics in a sample. a) All clusters. b) All complex clusters with an example of morphology evolution. c) All spherical clusters with an example of morphology evolution. d) Example of the exponential fit on two complex clusters and 3D rendering of their initial shape.

246 Saby et al. (2015b) noted that pores were closed for $Q < -0.2$ for STB model.
 247 For Nakasaki et al. (2006), closure is reached when $Q < -0.23$. Kakimoto et al.
 248 (2010) observed closure for $Q < -0.21$. Nevertheless, in the present case, many
 249 clusters are still open for such values. This difference can be explained by the

250 fact that in the cited papers, pores are either spherical or cylindrical whereas
251 real pores are considered in the present study, including complex shapes.

252

253 A large scatter in closure kinetics is observed. The influence of morphological
254 parameters is investigated to determine whether they can explain this scatter.

255 First, shape has an influence on pore closure kinetics. Figure 8.b presents
256 the closure kinetic of all complex clusters in a given sample. Figure 8.c presents
257 the closure kinetics of spherical clusters in the same sample. As spherical clusters
258 are smaller than complex clusters, the relative uncertainty in their volume
259 measurement is higher. Only their initial closure can be described as they reach
260 the minimal volume of 8 voxels for a higher normalized volume. For complex
261 clusters, the volume decreases exponentially with Q whereas spherical clusters
262 closure is more linear or quadratic with Q . This difference could be explained
263 by the fact that complex subpores fragment whereas spherical subpores remain
264 a unique entity during closure. Saby (2013) reported that fragmentation slows
265 down pore closure. In our case, fragmentation happens even in the early stage
266 of closure (Figure 7) when closure rate is still high and it rather seems that the
267 kinetics slow down when the biggest subpore of the cluster fragments in several
268 subpores of similar sizes.

269

270 An accurate description of the volume evolution of complex clusters is more
271 important than for spherical clusters since complex clusters are the largest and
272 the most detrimental for mechanical properties. We propose to describe complex
273 cluster closure kinetics using an exponential relation with the hydrostatic inte-
274 gration. For each complex cluster, the following formula was fitted, determining
275 an exponential coefficient A per cluster.

$$\frac{V}{V_0} = \exp(AQ) \quad (6)$$

276 Q being negative in compression and for positive values of A , this curve
277 decreases with cumulated strain. Figure 8.d shows the fitting curve for two

278 complex clusters. The higher the value of A , the faster the closure kinetics of
279 the pore.

280 The proposed fit can be expressed by the following derivative form:

$$\frac{\dot{V}}{V} = A T_X \dot{\epsilon} \quad (7)$$

281 It is the same expression as the analytical volume evolution of a spherical
282 pore in a linear viscous matrix calculated by Budiansky et al. Budiansky et al.
283 (1982) where $A = 9/4$.

284 The fit proposed here tends to slightly overestimate initial closure and un-
285 derestimate final closure but enables cluster closure ability to be easily sorted.
286 The exponential coefficient is determined for all complex pores in the 9 cylinder
287 samples. If the coefficient of determination r^2 is lower than 0.95, the pore is
288 not taken into account. Such low coefficient of determination occurs for pores
289 that are located in the zone of positive triaxiality or close to it. When triaxi-
290 ality becomes positive, the hydrostatic integration which was decreasing starts
291 to increase whereas pore volume continues to decrease before increasing. Mis-
292 fits also result from low negative triaxiality. In that case, cluster volume keeps
293 decreasing whereas hydrostatic integration decreases slower due to low nega-
294 tive triaxiality. It results in a too high negative slope compared to those of
295 the exponential formula. These observations show the limitation of hydrostatic
296 integration in the description of loading path.

297 In total 243 exponential coefficients A are fitted on closure kinetics of real
298 complex clusters. Distribution of all the values obtained is presented on Fig-
299 ure 9.a.

300 Different parameters are tested to draw possible correlations between the
301 exponential coefficients of a cluster and morphological and mechanical param-
302 eters. Some results are presented on Figures 9.b-d, all coefficients fitted for
303 complex clusters in cylindrical compressed samples are gathered on this figure.

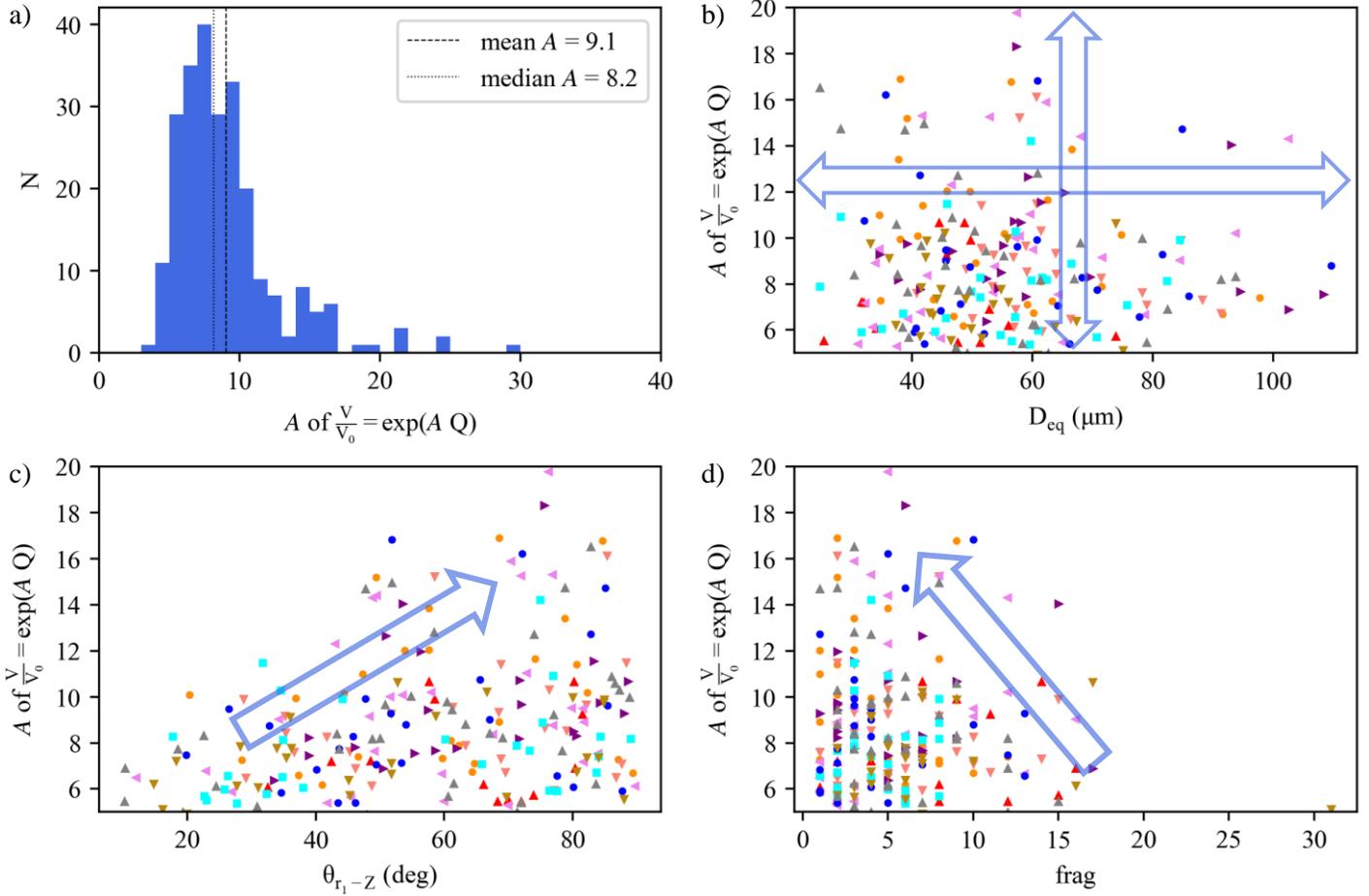


Figure 9: Study of the exponential coefficient A . a) Distribution of the values of A fitted on 243 closure kinetics of complex clusters. b-c) Dependency of exponential coefficient to several morphological parameters: equivalent diameter (b), orientation (c) and initial fragmentation (d).

304 The following observations can be made:

- 305 • A correlation between A and the equivalent diameter D_{eq} is not obvious

306 and thus, in the range of volume tested, pore closure does not depend on
307 pore size. This observation is consistent with results of Saby (2013) and
308 Chen and Lin (2013).

- 309 • Higher values of A are reached for clusters with a low initial fragmentation.
310 In this case, interaction between individual subpores in a cluster would
311 not be beneficial for closure. This is consistent with the results of Saby
312 (2013) who observed that fragmentation slows down the closure kinetics.
- 313 • A reaches the highest values for clusters oriented perpendicularly to the
314 deformation direction. This result is consistent with those of Saby (2013)
315 where faster closure was observed for ellipsoids compressed along their
316 smallest semi-axis (so compression direction forms an angle of 90° with the
317 longest semi-axis). It is also consistent with the model of Feng and Cui
318 (2015) which predicts faster closure for ellipsoids that have a low dimen-
319 sion in the compression direction. The orientation effect is accentuated by
320 large aspect ratios (r_1/r_3). Here, only orientation is taken into account
321 and not the ellipsoid aspect ratio. Thus, pores with low aspect ratio can be
322 oriented perpendicularly to the compression and result in a low A value.
323 Note that the notions of orientation and aspect ratio are more difficult
324 to define for a real tortuous pore than for an ellipsoid. If the pore is
325 branched, with arms in different directions, global orientation of the pore
326 does not faithfully represent the different arms. It would be more relevant
327 to adopt a more local description of the pore orientation.

328
329 As a conclusion, complex cluster closure kinetics are mostly influenced by
330 the initial fragmentation of clusters and their orientations. It is difficult to
331 determine the initial fragmentation and orientation of pores in an industrial
332 plate prior to rolling. Thus, we propose a new closure model expressed by
333 Equation 6 with a fixed value of $A = 9.1$ for all clusters. This phenomenological
334 description represents the average closure behavior of studied complex clusters
335 and captures the closure kinetics induced by fragmentation.

336 *4.5. Effects of temperature and compression speed on closure kinetics*

337 The previous results show data acquired during the compression of samples
338 at different temperatures (455, 480 and 520°C) and speeds (100, 500 and 1000
339 $\mu m.s^{-1}$). FEM simulations have shown that the local loading paths, described
340 by the evolution of stress triaxiality as a function of the cumulated strain, are
341 not influenced by the temperature or speed Gravier et al. (under revision -
342 PROTEC-D-21-02245). Thus, closure kinetics were considered independently
343 of the compression conditions.

344 Figure 10 shows the distribution, under the shape of a boxplot, of the ex-
345 ponential coefficients in each studied sample (ends of the whiskers correspond
346 to the largest points that fall within a distance of less than 1.5 times the in-
347 terquartile range over the third quartile and under the first quartile). If the
348 experimental conditions used to test a sample act in favor of pore closure, the
349 distribution would be shifted toward higher A values. The figure shows that dis-
350 crepancies between standard conditions and tests with different temperatures
351 and speeds are less significant than discrepancies between samples tested with
352 same standard conditions. It means that temperature and compression speed
353 have a second order influence on pore closure compared to cluster morphology
354 such as orientation or fragmentation.

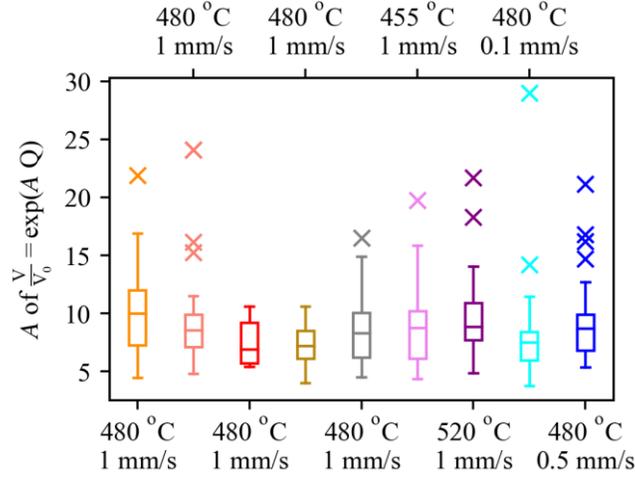


Figure 10: Boxplots of the distribution of exponential coefficient for the different samples.

355 5. Conclusions

356 In this work we were able to study porosity closure based on the observa-
 357 tion of pores during their deformation using X-ray microtomography. These
 358 interrupted in situ tests were performed under conditions representative of hot
 359 rolling and the closure of several hundreds of real pores was followed. Pores
 360 were studied at two scales (subpores and clusters) and as a function of their
 361 shape (spherical, rod, equiaxed and complex). Based on this work, the follow-
 362 ing conclusions can be drawn:

- 363 • Complex clusters are bigger in size and represent the largest part of the
 364 porosity compared to pores with other shapes. The accuracy of the overall
 365 porosity prediction is mainly linked to the ability to properly describe
 366 complex subpores closure.
- 367 • During closure, complex subpores fragment into smaller subpores with
 368 simpler shapes.

- 369 • Closure kinetics strongly depend on cluster shape. Closure kinetics of
370 complex clusters can be approximated by an exponential function of the
371 hydrostatic integration whereas a linear or quadratic dependency on the
372 hydrostatic integration is more suitable in the case of spherical clusters.
- 373 • For complex clusters, closure is influenced by the principal orientation and
374 the initial fragmentation of the cluster. Clusters oriented perpendicularly
375 to the compression direction and with low initial fragmentation are easier
376 to close.
- 377 • Based on experimental data analysis, a new closure model is proposed:
378 $V/V_0 = \exp(9.1 Q)$. It only depends on the hydrostatic integration. It
379 aims at properly describing complex pore closure up to relative volume
380 far below 20% because pore dimensions are still large for such a remaining
381 volume.

382 This study proposes a prediction model for pore closure by fitting experi-
383 mental data. Nevertheless, many other models are available in the literature.
384 Some of these models will be tested in a future paper. These models can be
385 computed using the present data collected with X-ray microtomography and
386 with the local mechanical fields provided by simulations.

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393 **References**

394 Budiansky B, Hutchinson J, Slutsky S. Void Growth and Collapse in Viscous
395 Solids. *Mechanics of Solids* 1982;1(3):13–45.

- 396 Chbihi A, Bouchard PO, Bernacki M, Pino Muñoz D. Influence of Lode angle
397 on modelling of void closure in hot metal forming processes. *Finite Elements*
398 *in Analysis and Design* 2017;126(September 2016):13–25.
- 399 Chen Kb, Yang Y, Liu K, Shao G. Simulation of void defect evolvment during
400 the forging of steel ingot. *Advanced Materials Research* 2010;97-101:3079–84.
- 401 Chen MS, Lin YC. Numerical simulation and experimental verification of void
402 evolution inside large forgings during hot working. *International Journal of*
403 *Plasticity* 2013;49:53–70.
- 404 Couper MJ, Neeson AE, Griffiths JR. Casting Defects and the Fatigue Be-
405 haviour of an Aluminium Casting Alloy. *Fatigue & Fracture of Engineering*
406 *Materials & Structures* 1990;13(3):213–27.
- 407 Dantzig JA, Rappaz M. *Solidification: Revised and Expanded*. EPFL press,
408 2016.
- 409 Duva J, Hutchinson J. Constitutive potentials for dilutely nonlinear materials.
410 *Chemical Physics* 1984;3(July):228–34.
- 411 Fang H, Versteyleen CD, Zhang S, Yang Y, Cloetens P, Ngan-tillard D. Au-
412 tonomous filling of creep cavities in Fe-Au alloys studied by synchrotron X-ray
413 nano-tomography. *Acta Materialia* 2016;121:352–64.
- 414 Feng C, Cui Z. A 3-D model for void evolution in viscous materials under large
415 compressive deformation. *International Journal of Plasticity* 2015;74:192–212.
- 416 Gravier P, Mas F, Barthelemy A, Boller E, Salvo L, Lhuissier P. Pore clo-
417 sure in thick aluminum plate: from industrial hot rolling to individual pore
418 observation. *Journal of Materials Processing Technology under revision -*
419 *PROTEC-D-21-02245*;
- 420 Gurson AL. Continuum Theory of Ductile Rupture by Void Nucleation and
421 Growth: Part I—Yield Criteria and Flow Rules for Porous Ductile Media.
422 *Journal of Engineering Materials and Technology* 1977;99(1):2.

- 423 Kakimoto H, Arikawa T, Takahashi Y, Tanaka T, Imaida Y. Development of
424 forging process design to close internal voids. *Journal of Materials Processing*
425 *Technology* 2010;210(3):415–22.
- 426 Kishimoto K, Soboyejo WO, Knott JF, Smith RA. A numerical investigation of
427 the interaction and coalescence of twin coplanar semi-elliptical fatigue cracks.
428 *International Journal of Fatigue* 1989;11(2):91–6.
- 429 Llanos JM, Santisteban V, Demurger J. Improvement of central soundness in
430 long products from a through process control of solidification and reheating
431 and rolling. Technical Report; European Commission - Research Fund for
432 Coal and Steel; 2008.
- 433 Murakami Y, Endo M. Effects of defects, inclusions and inhomogeneities on
434 fatigue strength. *International Journal of Fatigue* 1994;16(3):163–82.
- 435 Nakasaki M, Takasu I, Utsunomiya H. Application of hydrostatic integration
436 parameter for free-forging and rolling. *Journal of Materials Processing Tech-*
437 *nology* 2006;177(1-3):521–4.
- 438 Park C, Yang D. A study of void crushing in large forgings I: Bonding mechanism
439 and estimation model for bonding efficiency. *Journal of Materials Processing*
440 *Technology* 1996;57(1-2):129–40.
- 441 Saby M. Compréhension et modélisation des mécanismes de refermeture de
442 porosité dans les procédés de mise en forme des métaux à chaud. Ph.D.
443 thesis; Mines ParisTech; 2013.
- 444 Saby M, Bouchard PO, Bernacki M. A geometry-dependent model for void
445 closure in hot metal forming. *Finite Elements in Analysis and Design*
446 2015a;105:63–78.
- 447 Saby M, Bouchard PO, Bernacki M. Void closure criteria for hot metal forming:
448 A review. *Journal of Manufacturing Processes* 2015b;19(2):239–50.

- 449 Tanaka M, Ono S, Tsuneno M. Factors contributing to crushing of voids during
450 forging. *J Jpn Soc Technol Plast* 1987;27(306):852–9.
- 451 Wang X, Dong X. A void evolution model accounting for stress triaxiality, Lode
452 parameter and effective strain for hot metal forming. *International Journal*
453 *of Mechanical Sciences* 2020;168(November 2019). doi:10.1016/j.ijmecsci.
454 2019.105309.
- 455 Wu X, Schlangen E, van der Zwaag S. Linking Porosity to Rolling Reduction
456 and Fatigue Lifetime of Hot Rolled AA7xxx Alloys by 3D X-Ray Computed
457 Tomography. *Advanced Engineering Materials* 2012;14(7):457–63.
- 458 Zhang Q, Niu L, Liang Z, Cao M, Zhou T. A porosity closure model consid-
459 ering stress triaxiality ratio and Lode stress parameter. *Journal of Materials*
460 *Processing Technology* 2020;286(May):116824.
- 461 Zhang XX, Cui ZS, Chen W, Li Y. A criterion for void closure in large ingots dur-
462 ing hot forging. *Journal of Materials Processing Technology* 2009;209(4):1950–
463 9.