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## ► To cite this version:

Gaetan Boissonneau, Andrea Tommasi, Fabrice Barou, Marco A. Lopez-Sanchez, Maurine Montagnat. Dynamic recrystallization and mechanical behavior of Mg alloy AZ31: Constraints from tensile tests with in-situ EBSD analysis. *Comptes Rendus. Mécanique*, 2024. hal-04388880v1

**HAL Id: hal-04388880**

**<https://hal.science/hal-04388880v1>**

Submitted on 15 Jan 2024 (v1), last revised 6 Sep 2024 (v2)

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# Dynamic recrystallization and mechanical behavior of Mg alloy AZ31: Constraints from tensile tests with in-situ EBSD analysis

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## Abstract

We conducted tensile tests on AZ31 samples with in-situ SEM-EBSD acquisition at 250°C and  $10^{-3} \text{ s}^{-1}$  to study the evolution of dynamic recrystallization and its effect on the mechanical behavior. To investigate the entire deformation range up to failure at 65-67% engineering strain, stepwise experiments were conducted with in-situ EBSD acquisition at 2-5% strain intervals and repolishing of the samples at 15% strain intervals. All experiments show a consistent mechanical behavior characterized by a transition from hardening to quasi-steady state at  $\sim 10\%$  strain, followed by softening for strains greater than 35%. The bulk intragranular misorientation, quantified by the mean kernel average misorientation, increases steadily. Analysis of the EBSD maps shows that the quasi-steady state is associated with dominant dislocation reorganization (polygonization), whereas the onset of softening is linked to a net and continuous increase in the recrystallized fraction. In situ EBSD mapping documents the local discontinuous nature of continuous dynamic recrystallization: (1) nucleation of strain-free grains by bulging and subgrain rotation, (2) grain growth, (3) re-accumulation of dislocations substructures and formation of new nuclei. Analysis of the textures shows that dynamic recrystallization slows down rotation and strengthening of the textures. The observed steady state and softening behaviors result therefore from both geometrical (texture-induced) and microstructural (dislocation-related) softening due to dynamic recrystallization.

*Keywords:* Dynamic recrystallization, softening, texture, hexagonal

## 22 1. Introduction

23 Dynamic recrystallization (DRX) shapes the mechanical properties and texture of polycrystalline materials during deformation at high temperature. It produces bulk softening by a series of processes, which occur at grain and lower scales (cf. reviews by Sakai et al.; Humphreys et al. [1, 2]). Local grain-to-grain strain incompatibility results in stress concentrations, which lead to nucleation by grain boundary migration (bulging), development of low-angle boundaries by reorganization of the dislocations within a grain, or both. The newly formed grains then grow as function of the local gradients in dislocation density, and the energy and properties of grain boundaries. If grain growth is slower than nucleation, DRX decreases the average grain size. DRX also changes the texture evolution as the newly formed grains inherit different orientations from the parent grains and may thereby affect the mechanical behavior. However, the local nature and complex interactions between these processes hinder the establishment of clear relations describing the impact of DRX on the mean properties of a polycrystal despite more than five decades of research efforts.

39 Tests with quasi in-situ Electron Backscatter Diffraction (EBSD) monitoring of the microstructure evolution allow combining: (1) local observations of the physical processes involved in DRX, (2) quantitative analysis of the microstructure changes at the polycrystal scale, and (3) the evolution of the mechanical behavior with increasing strain. Here we present tensile tests on AZ31 magnesium alloy samples covering a large strain range, up to 67% engineering strain, for fixed strain and temperature conditions, selected to maximize DRX. In addition to the evident industrial interest for a better understanding of DRX in magnesium alloys [3], the hexagonal symmetry and strong viscoplastic anisotropy of magnesium result in local strain incompatibility and stress concentrations, which trigger DRX. AZ31 alloys are therefore an excellent analog material to study the impact of DRX in materials in which it is an essential process during high-temperature deformation, but for which experiments using in-situ EBSD monitoring are not possible, like ice [4, 5] or rocks [6, 7].

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56 In the present study, we analyze the mechanical and bulk microstructural  
57 data (intragranular misorientation, grain size, and recrystallized fraction)  
58 for seven samples deformed to final engineering strains ranging from 32%  
59 to 67%. We document the different processes involved in DRX based on  
60 sequential misorientation maps. We then compare the observed texture evo-  
61 lution with predictions of viscoplastic self-consistent simulations to constrain  
62 how the microstructural and textural changes produced by DRX impact the  
63 mechanical behavior.

## 64 **2. Methods**

### 65 *2.1. Starting material*

66 Bone-shaped tensile samples (Supplementary Material Fig. S1) were cut  
67 from a cylindrical rod of commercially pure hot-extruded AZ31 magnesium  
68 alloy by electric discharge machining at 30-35° to the initial extrusion di-  
69 rection. This results in a strong initial texture characterized by a girdle of  
70  $\langle c \rangle$  axes at 55-60° to the traction axis, which inhibits twinning during  
71 elongation. Before deformation, the samples were annealed at 400°C for 24  
72 hours and polished with: #2400 and #4000 mesh grits, 6, 3, and 1  $\mu\text{m}$   
73 oil-based diamond suspensions and Oxide Polishing Suspension (OPS). Ul-  
74 trasonic ethanol baths were used to clean the surface between the different  
75 polishing steps and before EBSD analysis.

### 76 *2.2. In-situ tensile testing*

77 Tensile tests were conducted inside the CamScan X500-FE CrystalProbe  
78 SEM-EBSD chamber using a NewTec Scientific MT1000 high temperature  
79 tensile rig. Samples were heated to 250°C ( $T/T_f = 0.58$ ) and deformed at a  
80 constant velocity of 7  $\mu\text{m s}^{-1}$  ( $10^{-3} \text{ s}^{-1}$  in strain rate at 0% strain). The tem-  
81 perature is controlled using thermocouples located under the sample heads  
82 and in the center of the gauge section. Engineering strains are calculated  
83 based on a 7 mm initial length. Stress data are corrected for the reduction  
84 in section with increasing strain by assuming conservation of volume and  
85 uniform deformation of the sample.

86  
87 The deformation was halted at engineering strain intervals varying between  
88 2 to 5% for EBSD mapping. Interruption of deformation at elevated tem-  
89 peratures disturbs the macroscopic mechanical response of the specimens (cf.

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10 section 3.1). Two strategies were used to minimize the perturbation: (i) halt-  
11 ing deformation while holding the sample at 250°C, which results in stress  
12 relaxation during mapping, and (ii) rapidly cooling the sample to 150°C upon  
13 reaching the target strain and keeping it at 150°C during the EBSD acquisi-  
14 tion, which produces a stress peak due to thermal contraction.  
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17 Out-of-plane deformation during the experiment reduces the indexing rate of  
18 EBSD maps, particularly around grain boundaries and triple junctions, where  
19 dynamic recrystallization processes are expected to occur. By adjusting the  
20 averaging parameter of the Kikuchi pattern acquisition, i.e. increasing the  
21 number of Kikuchi patterns acquired in each measurement, we were able to  
22 obtain high quality EBSD maps for up to 15% cumulated engineering strain  
23 without repolishing. To map recrystallization processes at larger strains,  
24 samples were repolished at 15% strain intervals. This strategy produces a  
25 continuous dataset up to engineering strains of 67%.  
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### 29 *2.3. EBSD acquisition and post-processing*

30 EBSD was acquired on a fixed small domain in the central part of the sam-  
31 ple during each experiment. In addition, at the end of each experiment,  
32 post-mortem EBSD maps of larger area were acquired after repolishing the  
33 samples. The acquisition conditions, area and step sizes for all maps are  
34 summarized in Supplementary Material Table T1.  
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38 The open-source MTEX MATLAB toolbox v5.6.1 ([https://github.com/mtex-  
39 toolbox/mtex](https://github.com/mtex-toolbox/mtex)) has been employed to treat and analyze the EBSD data. Mea-  
40 surements with a mean angular deviation higher than 1.3° were neglected.  
41 Grain boundaries were identified using a 7.5° misorientation threshold and  
42 smoothed (two iterations). Grains defined by less than four pixels were  
43 dropped out from the dataset. EBSD data was smoothed using a half-  
44 quadratic filter within each grain.  
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49 We imaged and quantified the evolution of the microstructure based on: (1)  
50 the intragranular misorientation, (2) the grain sizes, and (3) the recrystallized  
51 fraction. The intragranular misorientation produced by the accumulation of  
52 geometrically necessary dislocations (GND), was estimated based on: (1)  
53 the kernel average misorientation (KAM), which is the average value of the  
54 misorientation between a pixel and its neighbors, (2) the grain orientation  
55 spread (GOS), which is the mean deviation of the local orientations relative  
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127 to the mean orientation of the grain, and (3) the misorientation (axis and  
128 intensity) to the mean orientation of the grain (Mis2mean). The first order  
129 KAM data was used if not specified otherwise. KAM and Mis2mean maps  
130 were used to document subgrain boundaries and subgrain structures within  
131 a grain, respectively. For comparing KAM data over the entire dataset, the  
132 higher resolution EBSD maps were down-sampled to match the coarser step  
133 size ( $2\ \mu\text{m}$ ). Grain sizes were quantified by the diameter of a circle with the  
134 same area as the grain (i.e. equivalent diameter).

135  
136 In the present study, recrystallized grains are identified based on either the  
137 GOS, which is a scalar measure of the intragranular misorientation, or the  
138 grain size. The justification for these criteria is that DRX creates new grains  
139 with initially small sizes and very low dislocation densities. Both criteria  
140 have limitations. Using solely a GOS threshold has the limitation of includ-  
141 ing statically recrystallized grains produced by the pre-deformation annealing  
142 as well as grains poorly oriented for dislocation glide that deform slowly in  
143 the DRX population. For instance, the undeformed samples have recrystal-  
144 lized fractions determined using this criterium of  $>90\%$ . Using a grain size  
145 threshold, this problem is avoided, but this criterium suffers from apparent  
146 2D section problems. One could therefore conclude that the best choice is to  
147 use a joint threshold. However, analyzing the RX fraction defined based each  
148 of these criterium separately allows to highlight different aspects of the RX  
149 process. The GOS threshold allows to discriminate newly formed RX grains  
150 with low dislocation densities from those that have a dislocation substructure  
151 due to either nucleation by polygonization or deformation subsequent to RX.  
152 The grain size threshold includes nuclei formed by any processes but keeps no  
153 memory of fast-growing RX grains; it allows to probe the grain growth rate.  
154 The absolute values of the two thresholds are chosen empirically. We present  
155 results for a GOS threshold of  $1^\circ$  and an equivalent grain diameter of  $20\ \mu\text{m}$ ,  
156 based on the analysis of the KAM maps and grain size distributions. Dif-  
157 ferent grain size and GOS threshold values result in different recrystallized  
158 fractions but define similar evolution trends (see Supplementary Material  
159 Fig. S2).

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160 **3. Results**

161 *3.1. Bulk mechanical behavior*

162 All experiments display a consistent mechanical behavior well illustrated by  
163 the true stress/engineering strain curves for the two samples deformed up to  
164 the highest strain (Fig. 1). Sample H (purple curve in Fig. 1) was deformed  
165 outside the SEM without interruption until failure to be used as a reference.  
166 The true stress/engineering strain curve displays an elastic loading phase of  
167 up to 2% strain, followed by a hardening stage up to 10% strain. From 10%  
168 to ~30% strain, it exhibits a quasi-steady state behavior, which is followed  
169 by softening up to ductile failure at 63% strain. Optical inspection of the  
170 evolution of the sample shape with increasing strain did not document any  
171 progressive necking, but a catastrophic development of a semi-brittle shear  
172 band at the end of the experiment (see Supplementary Material Fig. S3).

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174 The behavior of Sample X is representative of the mechanical response of the  
175 experiments in which the deformation was halted for in-situ EBSD mapping.  
176 Each interruption induced a stress relaxation, which is manifested as a verti-  
177 cal drop in the stress/strain curve. Three distinct deformation experiments  
178 (0% to 35%; 35% to 47%; 47% to 67%) were performed sequentially on this  
179 sample, with unloading and repolishing of the sample surface between the  
180 experiments. The true stress/engineering strain curves of the three experi-  
181 ments were assembled to construct the curve shown by the blue line in Fig.  
182 1. The stress relaxation at 10% strain is a consequence of a reboot of the  
183 tensile rig software during the experiment due to a technical issue.

184  
185 The two strategies for EBSD acquisition presented in section 2.3 have been  
186 tried on this sample: the segments of the curve displayed with full lines in  
187 Fig. 1 present data for EBSD acquisition at 150°C, whereas those displayed as  
188 dashed lines show the data for EBSD acquisition at 250°C. Despite the stress  
189 relaxation due to the interruption of the traction during the EBSD acquisi-  
190 tion, the mechanical behavior of the samples with in-situ EBSD acquisition  
191 is consistent with that of sample H. It is characterized by elastic loading up  
192 to 2% strain, transition from hardening to quasi-steady state around 10%  
193 strain, and onset of softening around 35-37% strain. The stress peak at 35%  
194 strain for sample X is a consequence of thermal contraction of the sample  
195 as it was cooled down to room temperature. This might have caused some  
196 hardening and explain the 10 MPa offset of the mechanical data for strains

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197 >35%. For the other experiments, unloading and cooling were synchronized  
198 to avoid such stress peaks. Post-mortem optical analysis of the sample at  
199 the end of the experiment highlights a stronger out-of-plane deformation and  
200 incipient necking in a  $\sim 3$  mm wide domain at the center of the specimen (see  
201 Supplementary Material Fig. S3). In the experiments halted at lower strains,  
202 necking is not observed.

### 203 *3.2. Bulk microstructural evolution*

204 The increase in KAM values show a steady accumulation of lattice misorien-  
205 tation (i.e., of GNDs) throughout the deformation experiment for all samples  
206 (Fig. 2.a). The median increases quasi-linearly, but the KAM distribution  
207 becomes progressive more skewed, due to faster increase of the higher values  
208 relative to the median, marking the development of low angle grain bound-  
209 aries with progressively higher misorientations. Domains characterized by  
210 null KAMs are preserved over the entire deformation range. The data from  
211 the first experiment on Sample X show slightly higher rates of lattice mis-  
212 orientation accumulation than the remainder of the dataset. However, this  
213 difference may result from a sampling bias, as these data have been collected  
214 with a coarser step size than the remainder of the data (cf. Supplementary  
215 Material Table T1).

216  
217 The grain size shows a two-step evolution (Fig. 2.b). All parameters de-  
218 scribing the grain size distribution (median, first and third quartiles, and  
219 maximum excluding outliers) decrease with strain up to 35% engineering  
220 strain and remain almost constant afterwards. Median equivalent diameters  
221 evolve abruptly from  $>20 \mu\text{m}$  to  $\sim 7 \mu\text{m}$  at strains  $>35\%$ . Note, however,  
222 that this shift in behavior has not been observed within a continuous ex-  
223 periment, but between successive experiments. Moreover, due to the limited  
224 area of the domains analyzed during in-situ mapping, boundary grains could  
225 not be eliminated from the dataset, biasing the estimation of the equivalent  
226 diameters towards lower values.

227  
228 The RX fraction, that is, the area fraction of the map occupied by recryst-  
229 tallized grains, defined using a  $1^\circ$  GOS threshold (see section 2.3) is initially  
230 very high because of artifacts (Fig. 3.a). Indeed, undeformed samples have  
231 “statically” recrystallized fractions ranging between 88-91%. As deforma-  
232 tion proceeds, the recrystallized fraction drops quickly to low values ( $<5\%$ )



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10 233 as the annealed grains start to accumulate dislocations. Despite some vari-  
11 234 ability among samples, due to the small mapped areas, the recrystallized  
12 235 fraction remains stable, below 5% up to 35% engineering strain. A sudden  
13 236 increase in the RX fraction is observed at  $\sim 35\%$  strain. In the 35% to 48%  
14 237 interval the RX fraction ranges between 7 and 12%. Thereafter, it fluctuates  
15 238 between 5 and 8%, but the last strain interval is documented by a single  
16 239 experiment. Post-mortem analysis of sample H documents higher RX frac-  
17 240 tions of 17% - 20% with the highest fractions measured in the vicinity of the  
18 241 localized shear band.  
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23 243 Defining recrystallized grains based on a  $20\ \mu\text{m}$  equivalent diameter threshold  
24 244 (Fig. 3.b) results in low RX fractions ( $<5\%$ ) up to 10% strain. Data for  
25 245 strains between 10% and 35% strain show a higher dispersion, but the RX  
26 246 fraction remains below 10%. Similarly to the RX fraction defined based on  
27 247 the GOS criterium, a sudden increase in the RX fraction is observed at 35%  
28 248 strain. Both samples deformed to higher strains display a steady increase  
29 249 in recrystallized fraction up to the end of the experiments, but the rates of  
30 250 increase differ between the two experiments. The maximum RX fraction,  
31 251 observed for sample F at 47% engineering strain, is of 28%.  
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### 34 252 *3.3. Recrystallization processes*

35  
36 253 The evolution of the microstructure from the annealed state up to 12% strain  
37 254 is illustrated using KAM maps of sample X (Fig. 4). Before deformation,  
38 255 grains have smooth boundaries and sizes ranging between 30 to  $100\ \mu\text{m}$ .  
39 256 Due to the pre-deformation annealing, KAM values are mostly below  $1^\circ$ ,  
40 257 thus most grains are identified as recrystallized based on the GOS criterium.  
41 258 A few grains preserved scarce subgrain boundaries. At 6% engineering strain,  
42 259 most grains display significant intragranular misorientations and the RX frac-  
43 260 tion based on the GOS is reduced to 15% (Fig. 3.a). Accumulation of dis-  
44 261 locations and formation of subgrain boundaries is spatially heterogeneous,  
45 262 varying from grain to grain, but, in most cases, densities are stronger in the  
46 263 vicinity of grain boundaries (on average within  $<10\ \mu\text{m}$  of the boundary).  
47 264 Apparent (2D) grain sizes hardly evolved, but grain boundaries became more  
48 265 serrated and local misorientations are intensified, leading to a decrease of the  
49 266 RX fraction defined based on the GOS to 3-4% at 12% engineering strain.  
50 267 At strains  $>10\%$ , small grains ( $<10\ \mu\text{m}$ ) are observed locally, but these small  
51 268 grains do not always have GOS  $<1^\circ$ . In summary, up to  $\sim 10\%$  engineering  
52 269 strain, the evolution of the substructure due to progressive accumulation of  
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270 geometrically necessary dislocations is more pronounced than the changes in  
271 grain boundary geometry. This observation indicates that, under the present  
272 experimental conditions, dislocation mobility has faster kinetics than grain  
273 boundary mobility.

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275 Analysis of the microstructure between 23% and 38% engineering strain for  
276 Sample D documents continuation of the same trend (Fig. 5.a). The more  
277 strained is the sample, the more serrated are the grain boundaries. The  
278 progressive increase in the misorientation across subgrain boundaries is well  
279 illustrated in the Mis2mean maps (right panel of Fig. 5.a). A zoom in the  
280 abnormally coarse grain that occupies most of the lower part of the map  
281 (Fig. 5.b) shows that the strong orientation gradient across the central part  
282 of the grain is accommodated by a network of low-angle boundaries, 10-20  
283  $\mu\text{m}$  wide. This network became more complex and misoriented with strain  
284 but did not evolve into a well-defined grain boundary. Most grains still have  
285 sizes in the 30 to 100  $\mu\text{m}$  range, but small DRX grains are observed in all  
286 maps. The RX fraction based on GOS remains almost constant (Fig. 3.a),  
287 but that based on grain size slowly increases (Fig. 3b). The discrepancy  
288 between the two measures may be partially explained by the contribution of  
289 polygonization and subgrain rotation to DRX. This process is well illustrated  
290 by the DRX grain highlighted in green in Fig. 5.b, in which the new grain  
291 boundaries, formed by polygonization, enclose preexisting substructure. This  
292 process contrasts with nucleation by bulging, in which grain boundary mi-  
293 gration cleans the dislocation substructure, producing DRX grains with little  
294 internal misorientation.

295  
296 A marked switch in mechanical behavior, from quasi steady-state to soften-  
297 ing (Fig. 1), accompanied by stabilization of the previously decreasing grain  
298 size (Fig. 2.b) and a sudden increase in the recrystallized fraction (Fig. 3)  
299 is observed around 35% engineering strain. The evolution of the microstruc-  
300 ture after this transition is well illustrated by data for sample X between  
301 35% and 47% engineering strain (Fig. 6). In situ EBSD data for the 47% to  
302 65% strain interval provide similar observations. The grain size distribution  
303 is strongly bimodal with coarse grains (equivalent diameters  $\geq 100 \mu\text{m}$ ) and  
304 clusters of small DRX grains (equivalent diameters of 3-20  $\mu\text{m}$ ). Most DRX  
305 grains show little intracrystalline misorientation (GOS  $< 1^\circ$ ). In the 35% to  
306 65% strain interval, the DRX clusters show fast microstructural evolutions  
307 relative to the coarse grains. The RX fraction defined based on the grain

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308 size increases slowly by nucleation along the boundaries of the initial coarse  
309 grains, whereas that based on the GOS remains stable.

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311 Relative to lower strains, which were dominated by polygonization, the con-  
312 tribution of nucleation and grain boundary migration to the evolution of the  
313 microstructure increases. Evidence of nucleation by bulging (grain boundary  
314 migration) with closure of the bulge by a subgrain boundary is widespread.  
315 This process is illustrated in Fig. 6.b, in which two grains formed by DRX  
316 are highlighted. The kinetics of the nucleation in the two cases is apparently  
317 distinct: the grain highlighted in purple formed progressively, whereas the  
318 one highlighted in green formed suddenly between 41% and 44% strain onto  
319 a previously slowly migrating boundary. The in-situ EBSD observations for  
320 this strain range highlight the discontinuous nature of the processes at the  
321 grain scale. The zoom in Fig. 6.c documents the evolution of a DRX grain,  
322 which was tracked based on its position and crystallographic orientation.  
323 This DRX grain grew fast at the expenses of its highly deformed neighbors,  
324 from 35% to 41% strain, while maintaining its intragranular structure almost  
325 free of dislocation substructures. At 44% growth slowed and dislocations  
326 started to accumulate, as documented by the build-up of substructures with  
327 misorientations that continued to increase up to 47% strain. This sequence  
328 also documents the formation of new DRX grains along the boundaries of  
329 this grain after 44% strain.

### 330 3.4. *Texture evolution*

331 The initial texture is characterized by a girdle of  $\langle c \rangle$  axes oblique to the  
332 tensile axis. We quantified the texture evolution by: (1) the change in the  
333 angle between the normal to the  $\langle c \rangle$ -axis girdle and the tensile axis and  
334 (2) the intensity of the texture using a texture index proxy, the J-index (the  
335 integral of the squared orientation distribution function [8]). With increasing  
336 strain, the angle between the normal to the  $\langle c \rangle$ -axis girdle and tensile axis,  
337 initially at 30-35°, decreases (Fig. 7.a). However, the texture reorientation  
338 rate progressively slows down and a quasi-steady orientation of the normal  
339 to the  $\langle c \rangle$ -axis girdle at  $\sim 20^\circ$  to the tensile axis is attained after 35%  
340 engineering strain. The evolution of the orientation of the DRX grains (seg-  
341 mented based on the grain size) is, however, different. They initially display  
342 similar, but more dispersed, orientations than those of the parent grains,  
343 which control the bulk texture. However, after 35% strain, DRX grains have

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10 344 a texture characterized by a higher angle between the normal to the  $\langle c \rangle$ -  
11 345 axis girdle and the tensile axis (ca.  $24^\circ$  at strains  $>45\%$ ).  
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13 347 The texture intensity does not vary significantly with increasing strain, with  
14 348 an almost constant bulk texture index around 4 for the datasets obtained on  
15 349 large areas at the start or after the experiments (Fig. 7.b). Higher texture  
16 350 indexes are estimated based on the smaller maps collected in-situ during the  
17 351 experiments, but these data are biased by the presence of a few coarse grains  
18 352 that occupy large area fractions of these maps (cf. Figs. 4 to 6). The DRX  
19 353 grains texture shows, like many other quantities in the present experiments,  
20 354 a change around 35% engineering strain. Up to 35% strain, it is slightly  
21 355 more dispersed than the bulk texture. Its dispersion then increases up to  
22 356 40% strain and then it stabilizes at texture indexes between 2 and 2.5.  
23 357

24 358 To quantify the effect of DRX on the texture evolution, we compare the  
25 359 measured textures to that predicted for uniaxial tension using a polycrystal  
26 360 plasticity model, in which deformation is solely accommodated by dislocation  
27 361 glide: the viscoplastic self-consistent model (VPSC [9, 10]). The simulation  
28 362 was performed using the VPSC7 code [11] with a 2<sup>nd</sup> order linearization  
29 363 procedure, normalized critical resolved shear stresses (CRSS) for the basal  
30 364  $\langle a \rangle$ , prismatic  $\langle a \rangle$ , and pyramidal  $\langle a + c \rangle$  systems of 1, 40, and 40  
31 365 MPa with the EBSD maps from the annealed state of sample Y as a reference  
32 366 texture. The ratios between the CRSS of the different slip modes are derived  
33 367 from Zhang et al. (2021) [12]. Since the microstructural observations point  
34 368 to effective recovery, hardening is not implemented in these simulations. The  
35 369 initial texture is that measured for the starting material. VPSC models  
36 370 predict continuous concentration and reorientation of the texture, though  
37 371 at a progressively decreasing rate, towards parallelism between the normal  
38 372 to the  $\langle c \rangle$ -axis girdle and tensile axis (continuous lines in Fig. 7). The  
39 373 texture evolution documented in the present experiments clearly deviates  
40 374 from the predictions of these DRX-free models. DRX produces therefore  
41 375 both dispersion and reorientation of the texture.  
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## 51 376 **4. Discussion**

### 52 377 *4.1. Experimental limitations*

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55 378 The first limitation of the present study stems from the interruption of the  
56 379 deformation for in-situ acquisition of the EBSD data. Reducing the temper-

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380 ature produces thermal stresses in the sample, whereas keeping the sample at  
381 250°C during the mapping may result in partial annealing and thus recovery.  
382 Then, the in-situ EBSD map sizes had to be kept small to prevent significant  
383 annealing and are therefore too small to be representative volume elements.  
384 To mitigate that, larger dimension maps were acquired post-mortem for most  
385 samples. These wide-ranging maps are used as reference to estimate the rep-  
386 resentability of the in-situ maps. The large number of EBSD maps acquired  
387 in the present study (61, cf. Supplementary Material Table T1) further en-  
388 sures the statistical representability of the bulk microstructural parameters.  
389 For the mechanical response, the data from Sample H, deformed outside the  
390 SEM without interruption until failure, is used as a reference.

391  
392 Due to the deterioration of the surface, acquisition of data on a single surface  
393 is limited to 15% cumulated strain before the samples had to be re-polished.  
394 However, the data presented in section 3.3 show that such a strain interval is  
395 sufficient to document the local DRX evolution. The analysis of multiple tests  
396 with overlapping strain intervals allows to verify the reproducibility of these  
397 observations and the sample scale evolution. Yet, the onset of widespread  
398 nucleation/growth around 35% was not observed within a single experiment,  
399 but by comparing different experiments. Finally, EBSD providing a two-  
400 dimensional description of the microstructure at the sample surface, growth  
401 of grain nucleus out of the observation plane cannot be discriminated from  
402 nucleation on the plane.

#### 403 *4.2. DRX processes*

404 The present experiments document continuous DRX in AZ31. This process  
405 involves: (1) accumulation and reorganization of dislocations in low-angle  
406 boundaries networks, i.e., polygonization (Figs. 4 and 5) and (2) grain for-  
407 mation processes (Fig. 6), with nucleation by an association of bulging and  
408 subgrain rotation (Fig. 6.b) and growth of the RX grains by grain boundary  
409 migration (Fig. 6.c). In most cases, new RX grains initially show low dislo-  
410 cation densities, documenting the cleaning of the dislocation substructure by  
411 migrating boundaries (Fig. 6.c). However, as they grow, they re-accumulate  
412 dislocations and re-recrystallize (Fig. 6.c).

413  
414 The DRX fraction remains limited up to 35% strain. Onset of widespread nu-  
415 cleation is associated with (1) a marked change in mechanical behavior, from  
416 quasi steady-state to softening (Fig. 1), (2) stabilization of the previously

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417 decreasing grain size (Fig. 2.b), and (3) sudden increase in the recrystallized  
418 fraction (Fig. 3). We infer that this rather long incubation period is needed  
419 to construct the dislocation substructure. Analysis of the KAM maps shows  
420 that substructure formation is more active during the hardening and steady-  
421 state phases of the experiments (Figs. 4 and 5) than during the softening  
422 phase (Fig. 6). The long incubation period suggests that, at the present  
423 experimental conditions, recovery may have delayed the onset of recrystal-  
424 lization. However, even when nucleation is dominated by bulging, the bulges  
425 are closed by subgrain boundaries that evolve into grain boundaries (Fig.  
426 6.b), indicating that polygonization plays an important role in nucleation [5].

427  
428 Comparison between the experimental textures and the texture evolution  
429 predicted by VPSC simulations highlights that during DRX both polygo-  
430 nization and nucleation/growth processes slow down the reorientation of the  
431 texture towards parallelism between the main Burgers vector, here  $[\bar{1}2\bar{1}0]$ ,  
432 and the tensile direction (Fig. 7). The experimental textures show a con-  
433 stant orientation for all strains  $>35\%$ , characterized by a bulk orientation of  
434 the normal to the  $\langle c \rangle$ -axes girdle at  $\sim 20^\circ$  to the tensile axis. RX grains  
435 are systematically more oblique to the tensile axis, having on average the  
436 normal to the  $\langle c \rangle$ -axes girdle at  $\sim 24^\circ$  to the tensile axis for all strains  
437  $>45\%$ . These observations suggest a stronger effect of nucleation/growth on  
438 the texture evolution.

439  
440 The difference between the recrystallized fractions defined using the GOS or  
441 the grain size stems from the fact that the two thresholds focus on different  
442 aspects of the recrystallization process (Fig. 3). The GOS criterion only  
443 considers the intragranular dislocation density. Grains are identified as RX  
444 grains until they re-accumulate dislocations by deformation. The grain size  
445 criterium is more sensitive to grain growth. It also identifies as RX grains  
446 those formed by subgrain rotation, which may have dislocation structures  
447 inherited from the parent grains and hence GOS values  $>1^\circ$ . This might  
448 explain the slightly higher RX fractions determined using the grain size cri-  
449 terium below 35% strain. However, the steady increase in the RX fraction  
450 after 35% strain determined using the grain size criterium while the RX frac-  
451 tion defined based on the GOS remains stationary results from slow grain  
452 growth relative to nucleation and build-up of dislocation substructures in the  
453 present experiments. This is corroborated by the marked grain size reduction  
454 (Fig. 2.b).

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11 456 Nucleation/growth never spreads over the entire sample. It is localized, form-  
12 457 ing discontinuous network of small-grain clusters (Fig. 6.a). This suggests  
13 458 that nucleation is mainly controlled by plastic strain incompatibilities, re-  
14 459 laxing local stress concentrations. The higher density of grain boundaries  
15 460 within the clusters may also favor further recrystallization within them, as  
16 461 it will favor the formation of dislocation pile-ups [13]. The RX fraction de-  
17 462 fined based on the grain size increases slowly as DRX progresses along the  
18 463 boundaries of the initial coarse grains linking the RX clusters. Progressive  
19 464 increase of the RX fraction and DRX spreading over large domains of the  
20 465 sample is consistent with the fact that necking only develops at high strains  
21 466 and quickly evolves into localized shear and failure.

#### 25 467 *4.3. Impact of DRX on the mechanical behavior*

26 468 All samples show a consistent mechanical behavior characterized by a tran-  
27 469 sition at  $\sim 10\%$  strain from hardening to a quasi-steady state, followed by  
28 470 softening for strains  $> 35\%$ . This contrasts with the steady increase of the  
29 471 KAM at the sample scale (Fig. 2.a). As the KAM directly correlates with  
30 472 the GND density, such an increase points to a steady increase in the GND  
31 473 content, which could lead to hardening. However, in the present experiments,  
32 474 polygonization may explain increase in KAM associated with a steady-state  
33 475 mechanical response. Organization of the dislocations into low angle bound-  
34 476 aries and progressive increase of their misorientation by absorption of new  
35 477 dislocations allow for increase in the average KAM without decrease of the  
36 478 dislocation mean free path.

37 479  
38 480 Onset of widespread nucleation and grain boundary migration controlled by  
39 481 contrasts in dislocation density are, however, required to explain the tran-  
40 482 sition from steady-state to softening at strains  $> 35\%$ . These processes produce  
41 483 softening as the migrating grain boundaries clean the dislocation substruc-  
42 484 tures (Fig. 6). They also increase the grain boundary area. This may  
43 485 have two opposite effects: (1) to enhance the contribution of grain bound-  
44 486 ary processes to deformation or (2) to hinder dislocation glide by increase  
45 487 in the density of high-angle grain boundaries, which will hinder dislocation  
46 488 glide and produce pile-ups, leading to hardening [14, 15]. In the present  
47 489 experiments, we observe both (1) a faster microstructural evolution of the  
48 490 DRX clusters relative to the remainder of the samples (Fig. 6.a) and (2)  
49 491 re-recrystallization of DRX grains (Fig. 6.c). These observations point to

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492 faster nucleation rates in the RX domains favored by stress concentrations  
10 due to dislocation pile-ups [13]. We hypothesize that fast re-recrystallization  
11 of DRX grains may avoid hardening by Hall-Petch processes during DRX.  
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14 DRX also produces softening by slowing down the texture evolution and  
15 therefore avoiding (or reducing) the geometrical hardening relative to that  
16 expected in absence of recrystallization. The relative contributions of the  
17 microstructural and the textural effects of DRX to the observed softening  
18 may be estimated by comparison between: (1) the mechanical behavior ob-  
19 served in the experiments, (2) the mechanical behavior predicted using the  
20 VPSC approach described in section 3.4, and (3) instantaneous tensional  
21 stresses predicted using the same VPSC model for the textures measured  
22 at different strains (Fig. 8). This comparison highlights that the geomet-  
23 rical weakening due to a slower texture evolution in the experiments alone  
24 could explain the quasi steady-state bulk mechanical behavior between 10  
25 and 35% engineering strain, but cannot account for the bulk softening ob-  
26 served for strains >35%. The comparison also shows that the slower texture  
27 evolution in the experiments starts before evidence for widespread nucleation  
28 is observed. This suggests that polygonization may effectively slow down the  
29 texture evolution and produce thereby significantly bulk softening. VPSC  
30 predictions are dependent to the first order on the CRSS used for the differ-  
31 ent slip systems [9]. A less anisotropic single crystal behavior (lower contrast  
32 between the CRSS of the basal and prismatic and pyramidal systems) will  
33 result in slower texture evolution and weaker geometrical hardening without  
34 DRX, but qualitatively the results will be similar.  
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42 Although nucleation is localized, affecting relatively small volumes (RX area  
43 fractions <30%, Fig. 3.b) in the shape of spatially discontinuous clusters  
44 (Fig.6.a), it produces bulk softening of the sample by relaxation of local  
45 stress concentrations. Homogenization of the stresses within the sample by  
46 DRX could explain tensile deformation to high strains without necking. In  
47 contrast, the final deformation stages of sample H seem to indicate that  
48 development of a throughgoing recrystallization domain at the sample scale  
49 may result in strain localization, dramatic softening, and shear failure. The  
50 present data raise an additional question: What phenomenon does control  
51 the transition in macroscopic behavior between steady-state and softening: a  
52 critical RX fraction or a percolation threshold characterizing the continuity  
53 on a given scale (sample width) of the RX clusters?  
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530 **5. Conclusion**

531 All experiments show a consistent mechanical behavior characterized by tran-  
532 sition at  $\sim 10\%$  strain from hardening to a quasi-steady state, followed by  
533 softening for strains  $> 35\%$ , despite a steady increase in the KAM ( $\sim$  geo-  
534 metrically necessary dislocation density) at the sample scale. Analysis of the  
535 EBSD maps shows that the quasi-steady state mechanical behavior is mainly  
536 associated with reorganization of the dislocations (polygonization), whereas  
537 the softening is linked to widespread nucleation and growth.

538 In-situ EBSD mapping allows monitoring the evolution of the dynamic re-  
539 crystallization (DRX) process at both local and bulk (sample) scales. At  
540 the sample scale, the EBSD observations document a rather long incubation  
541 period up to  $35\%$  strain, which is dominated by dislocation accumulation  
542 and reorganization (polygonization), followed by widespread, but spatially  
543 heterogeneously distributed grain nucleation and growth. Closer analysis of  
544 the recrystallization (RX) clusters highlights the local discontinuous nature of  
545 DRX with nucleation by coupled bulging and subgrain rotation, grain growth  
546 and re-accumulation of dislocation substructures. At the studied experimen-  
547 tal conditions, grain growth is slow relative to nucleation and build-up of  
548 dislocation substructures in the present experiments, producing a marked  
549 average grain size reduction. Despite the complexity and local nature of the  
550 DRX processes, the dataset acquired on multiple specimens is remarkably  
551 coherent.

552 In the present experiments, DRX grains are concentrated in clusters. DRX  
553 produces local softening and avoids stress concentrations due to strain in-  
554 compatibility, since by cleaning the dislocation substructures and increasing  
555 the grain boundary area. Homogenization of the stresses within the sam-  
556 ple by DRX could explain the observed tensile deformation to high strains  
557 without necking. DRX also slows down the texture strengthening compared  
558 to modeling, since the RX grains are preferentially oriented oblique to the  
559 tension direction, thereby decreasing the geometrical hardening relative to  
560 that expected in absence of recrystallization.

561  
562 On a more technical note, the present results highlight the arbitrary nature  
563 of the criteria used for identifying RX grains and determining RX fractions.  
564 Use of grain orientation spread (GOS) and grain size threshold produce com-  
565 pletely different results, even in a relative framework, as these criteria empha-  
566 size different aspects of the recrystallization process: decrease in the intra-

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567 granular geometrically necessary dislocation density vs. grain size, which are  
568 controlled by the nucleation processes and the relative kinetics of nucleation,  
569 grain growth and re-accumulation of dislocation substructures.

570 **Acknowledgements**

571 This work was supported by the European Research Council (ERC) un-  
572 der the European Union Horizon 2020 Research and Innovation programme  
573 [grant agreement No 882450 – ERC RhEoVOLUTION]. We are grateful to  
574 Christophe Nevado from Géosciences Montpellier, for his support in the  
575 preparation of the EBSD samples. We thank Thomas Chauve for fruitful  
576 discussions. We also thank Juliette Grosset for help with final formatting of  
577 the paper.

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9 **References**

- 10  
11 579 [1] T. Sakai, A. Belyakov, R. Kaibyshev, H. Miura, J. J. Jonas, Dynamic  
12 580 and post-dynamic recrystallization under hot, cold and severe plastic  
13 581 deformation conditions, *Progress in Materials Science* 60 (2014) 130–  
14 582 207. doi:10.1016/j.pmatsci.2013.09.002.
- 15  
16  
17 583 [2] J. Humphreys, G. S. Rohrer, A. Rollett, *Recrystallization and Related*  
18 584 *Annealing Phenomena*, Newnes, 2017.
- 19  
20 585 [3] T. Al-Samman, G. Gottstein, Dynamic recrystallization during  
21 586 high temperature deformation of magnesium, *Materials Sci-*  
22 587 *ence and Engineering: A* 490 (1-2) (2008) 411 – 420. doi:DOI:  
23 588 10.1016/j.msea.2008.02.004.  
24 589 URL <http://www.sciencedirect.com/science/article/B6TXD-4RTW3TJ-8/2/71432008df>
- 25  
26  
27 590 [4] M. Montagnat, T. Chauve, F. Barou, A. Tommasi, B. Beausir, C. Fres-  
28 591 sengeas, Analysis of Dynamic Recrystallization of Ice from EBSD Ori-  
29 592 entation Mapping, *Frontiers in Earth Science* 3 (2015).
- 30  
31  
32 593 [5] T. Chauve, M. Montagnat, F. Barou, K. Hidas, A. Tommasi, D. Main-  
33 594 price, Investigation of nucleation processes during dynamic recrystal-  
34 595 lization of ice using cryo-EBSD, *Philosophical Transactions of the Royal*  
35 596 *Society A: Mathematical, Physical and Engineering Sciences* 375 (2086)  
36 597 (2017) 20150345. doi:10.1098/rsta.2015.0345.
- 37  
38  
39 598 [6] J. Urai, W. Means, G. Lister, Dynamic recrystallization of minerals,  
40 599 in: *Mineral and Rock Deformation: Laboratory Studies*, Geophysical  
41 600 *Monograph*, Hobbs, B.E. and Heard, H.C., 1986, pp. 161–200.
- 42  
43 601 [7] M. A. Lopez-Sanchez, A. Tommasi, W. B. Ismail, F. Barou,  
44 602 Dynamic recrystallization by subgrain rotation in olivine re-  
45 603 vealed by electron backscatter diffraction, *Tectonophysics* (2021)  
46 604 228916doi:<https://doi.org/10.1016/j.tecto.2021.228916>.  
47 605 URL <https://www.sciencedirect.com/science/article/pii/S0040195121001980>
- 48  
49  
50 606 [8] H.-J. Bunge, J.-S. Lecomte, *Texture Analysis in Materials Science* H.-J.  
51 607 Bunge, Tech. rep. (1982). doi:10.13140/RG.2.1.1721.1041.
- 52  
53  
54 608 [9] R. A. Lebensohn, C. N. Tomé, A self-consistent viscoplastic model: pre-  
55 609 diction of rolling textures of anisotropic polycrystals, *Mat. Sci. and En-*  
56 610 *gin.*, A 175 (1993) 71–82.

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2  
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6  
7  
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9  
611 [10] R. A. Lebensohn, M. I. Idiart, P. Ponte-Castañeda, P.-G. Vincent,  
612 Dilational viscoplasticity of polycrystalline solids with intragranu-  
613 lar cavities, *Philosophical Magazine* iFirst (2011) 1–30. doi:DOI:  
614 10.1080/14786435.2011.561811.
- 615 [11] C. Tomé, R. Lebensohn, Manual for VPSC7c, Los Alamos National Lab-  
616 oratory, [https://public.lanl.gov/lebenso/VPSC7c\\_manual.pdf](https://public.lanl.gov/lebenso/VPSC7c_manual.pdf) (2012).
- 617 [12] F. Zhang, M. Sun, B. Sun, F. Zhang, Y. Bai, Z. Liu, Temperature  
618 Dependency on the Microscopic Mechanism in the Normal Direction of  
619 Wrought AZ31 Sheet under Dynamic Compressive Behavior, *Materials*  
620 14 (23) (2021) 7436. doi:10.3390/ma14237436.
- 621 [13] P. Duval, F. Louchet, J. Weiss, M. Montagnat, On the role of long-  
622 range internal stresses on grain nucleation during dynamic discontinuous  
623 recrystallization, *Materials Science and Engineering: A* 546 (2012) 207–  
624 211. doi:10.1016/j.msea.2012.03.052.
- 625 [14] E. O. Hall, The Deformation and Ageing of Mild Steel: II Characteristics  
626 of the Lüders Deformation, *Proceedings of the Physical Society. Section*  
627 *B* 64 (9) (1951) 742. doi:10.1088/0370-1301/64/9/302.
- 628 [15] H. Adachi, H. Mizowaki, M. Hirata, D. Okai, H. Nakanishi, Mea-  
629 surement of Dislocation Density Change during Tensile Deforma-  
630 tion in Coarse-Grained Aluminum by *In-Situ* XRD Technique with  
631 Tester Oscillation, *Materials Transactions* 62 (1) (2021) 62–68.  
632 doi:10.2320/matertrans.L-M2020861.

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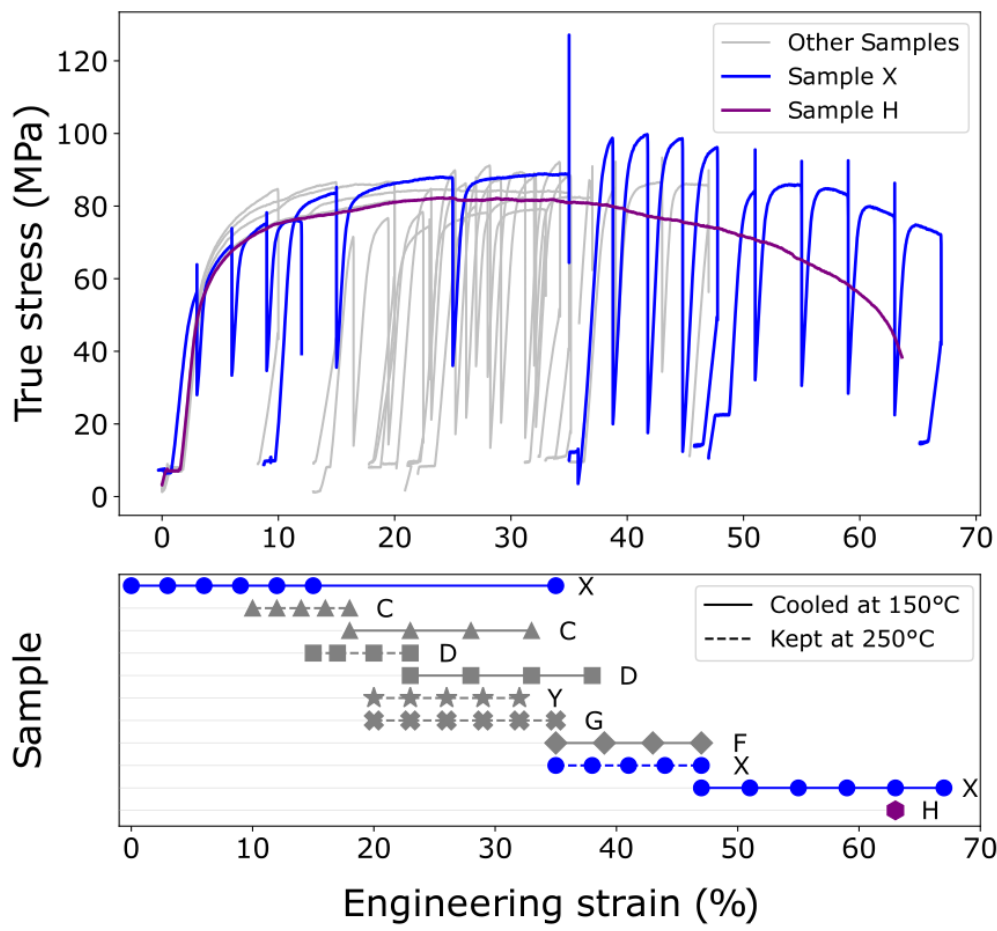


Figure 1: True stress/ engineering strain curves for all tests. Data for samples X and H, which cover the full strain range and are discussed in detail in the text, are highlighted in blue and purple, respectively. The lower panel details the experiments conducted on each sample: each line represents a continuous experiment with in-situ EBSD maps acquired without repolishing of the sample. Sample H was deformed without in-situ EBSD acquisition and is used as a reference.

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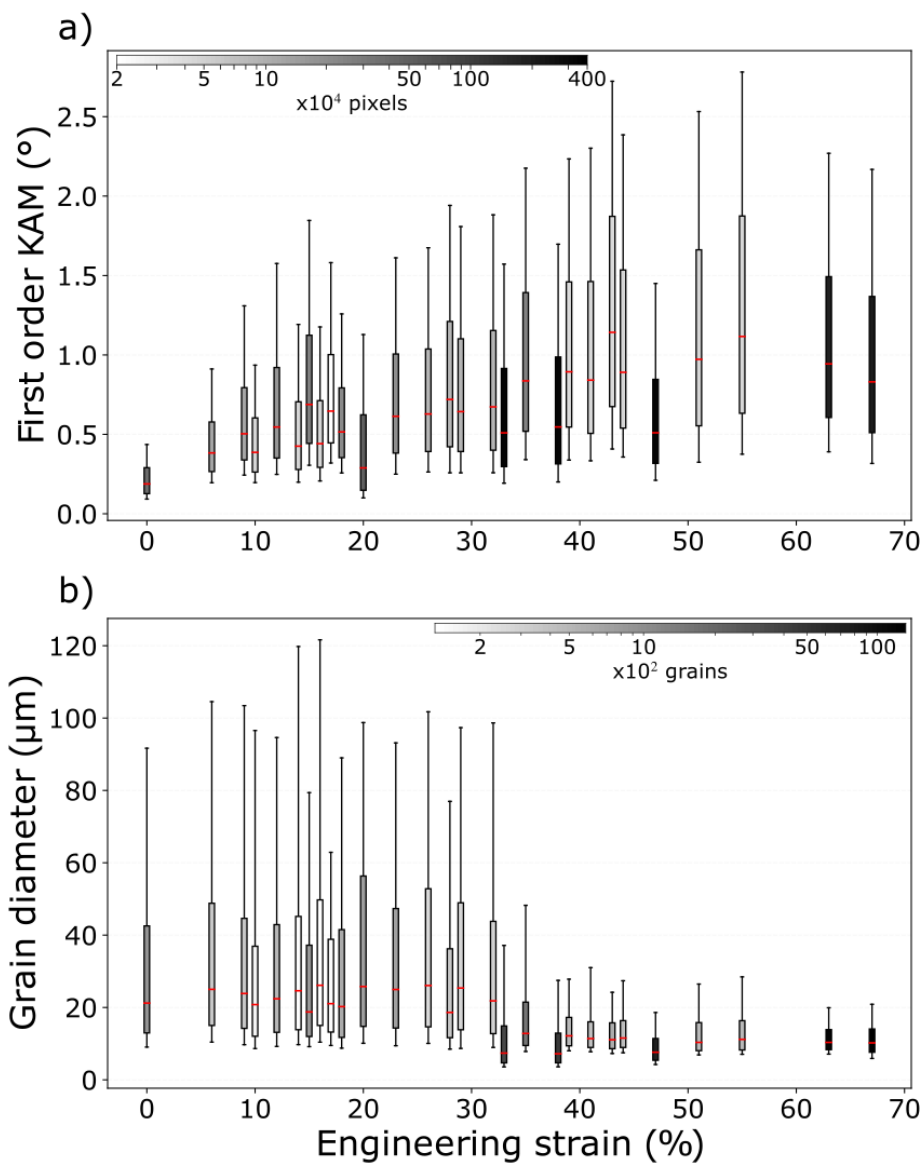


Figure 2: Evolution of the (a) intragranular misorientation and (b) grain size distributions with strain. Intragranular misorientation is defined by the 1st order Kernel Average Misorientation (KAM) and grain sizes, by the equivalent diameter. Data from multiple EBSD maps on a single or multiple samples at similar engineering strains were concatenated to increase the statistics. The boxes define the first and third quartiles, the median is highlighted in red, and the extrem bounds of the whiskers represent the 10th and 90th percentiles of the data. The box color is function of the number of data points in each distribution, with darker colors marking the data with best statistics (graphic legends on the top of each plot).

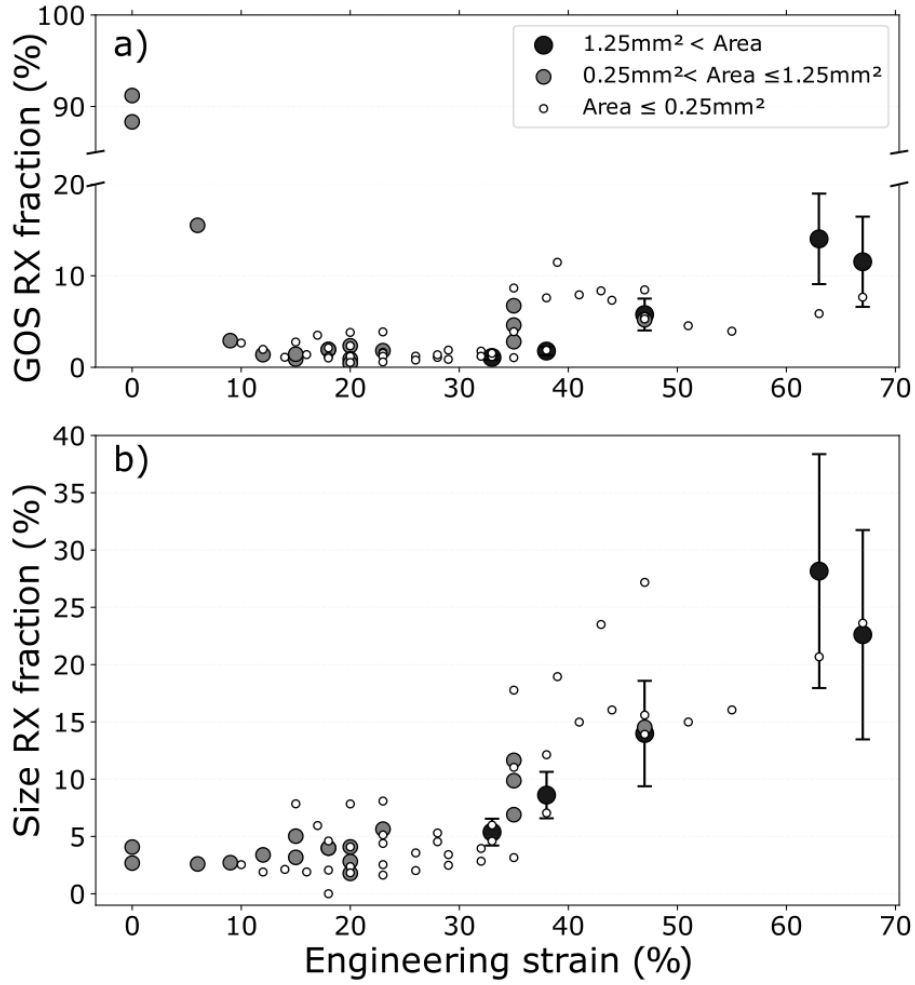


Figure 3: Evolution with strain of the recrystallized area fraction defined based on (a) a  $1^\circ$  GOS threshold and (b) a  $20 \mu\text{m}$  equivalent diameter threshold. Symbol sizes indicate the total area of the EBSD map. To estimate the spatial variability of the data and the representativity of the smaller in-situ maps, the large maps (area  $> 1.25 \text{ mm}^2$ ) have been re-sampled as multiple smaller maps with areas similar to that of the in-situ ones ( $\sim 0.2 \text{ mm}^2$ ); this allows to estimate the mean and standard deviation, represented by the errorbars, of the recrystallized area fraction distribution in these maps.

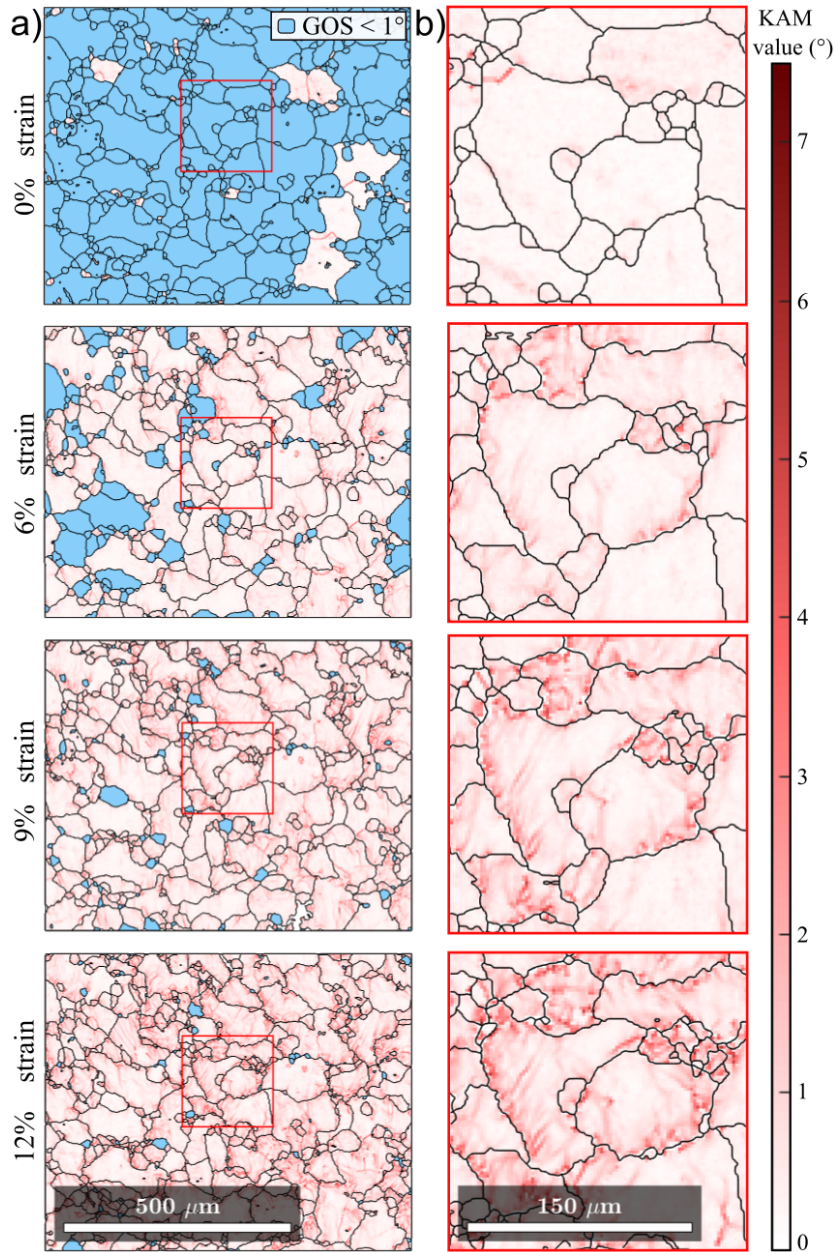


Figure 4: Microstructure evolution in the early stages of the experiments, illustrated by first order KAM maps obtained in situ at 0%, 6%, 9%, and 12% engineering strain for sample X. Grain boundaries, calculated using a  $7.5^\circ$  misorientation threshold, are represented as black lines. (a) Full size maps, in which the recrystallized grains identified using a  $1^\circ$  GOS threshold are coloured in blue. (b) Zoom on the area defined by the red square in (a); in these detail maps, KAM data for all grains are displayed, so that its evolution with strain may be followed.



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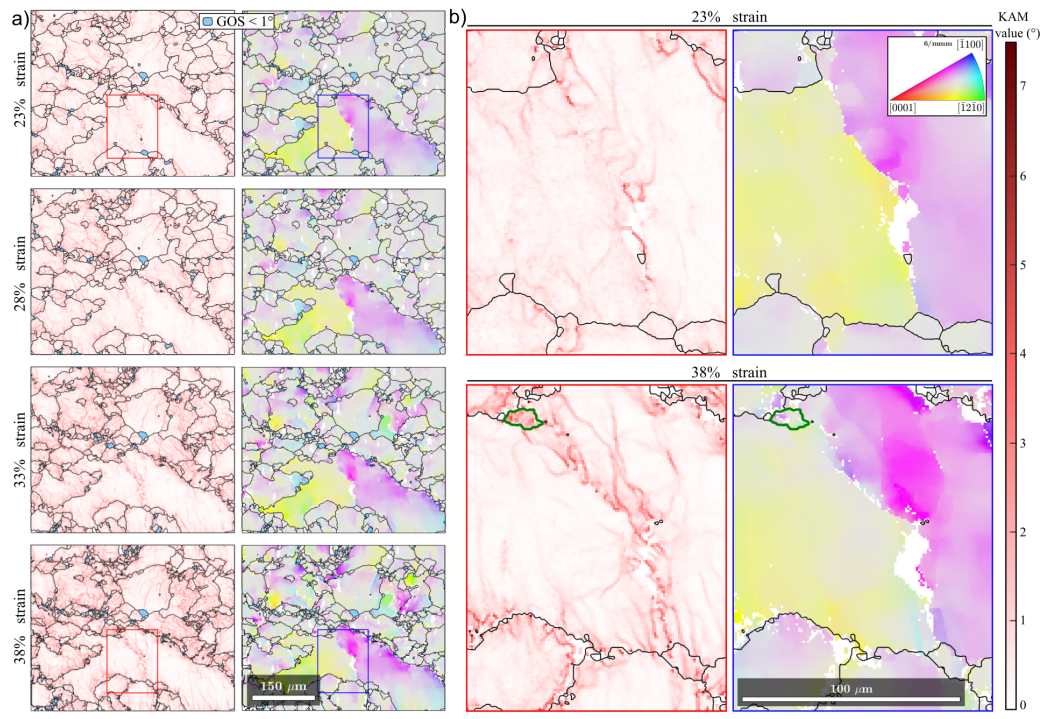


Figure 5: Microstructure evolution in the 23% to 38% strain interval, illustrated by in-situ first order KAM (left column) and mis2mean maps (right column) of sample D at 23%, 28%, 33%, and 38% engineering strain. Grain boundaries calculated using a  $7.5^\circ$  misorientation threshold are represented as black lines. (a) Full size maps, with recrystallized grains identified using a  $1^\circ$  GOS threshold coloured in blue. (b) Zoom on the area defined by the blue and the red rectangles in 23% and 38% engineering strain maps. A grain interpreted as formed by nucleation by subgrain rotation between 33% and 38% strain, is highlighted with a green boundary in the 38% strain detail maps, note its strong intragranular misorientation.

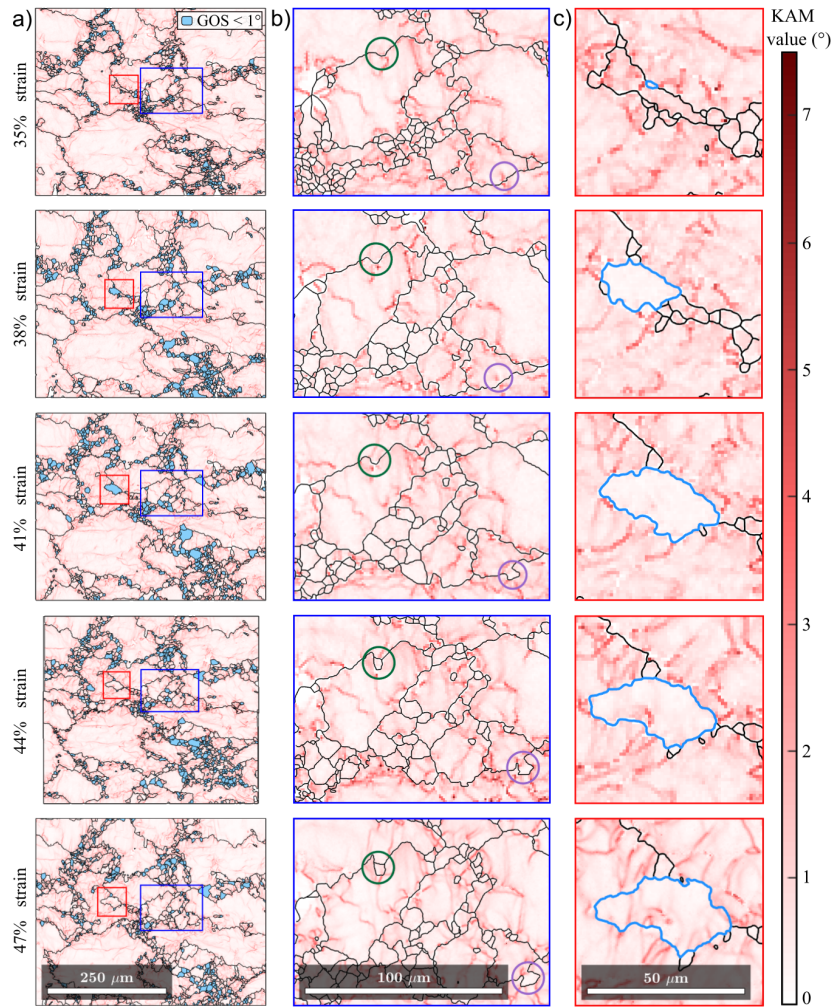


Figure 6: Microstructure evolution in the softening stage of the experiments, illustrated by KAM maps obtained in situ for sample X at 35%, 38%, 41%, 44%, and 47% engineering strain. All maps but that at 47% strain, were acquired with a  $1 \mu\text{m}$  step size; the map at 47% strain was acquired with a step size of  $0.5 \mu\text{m}$ . To keep absolute KAM values comparable over the entire strain range, the KAM order has been adjusted: first order KAM data is presented for all maps except for that at 47% strain, which displays second order KAM data. Grain boundaries calculated using a  $7.5^\circ$  misorientation threshold are represented by black lines. (a) Full size maps, in which recrystallized grains identified using a  $1^\circ$  GOS threshold are coloured in blue. (b) Zoom on the area defined by the blue rectangle in (a); dark green and purple circles are used to highlight areas showing nucleation events. (c) Zoom on the area defined by the red rectangle in (a), which displays the growth and re-accumulation of local misorientation in a recrystallized grain (highlighted in light blue) that is tracked using its location and crystallographic orientation from map to map.

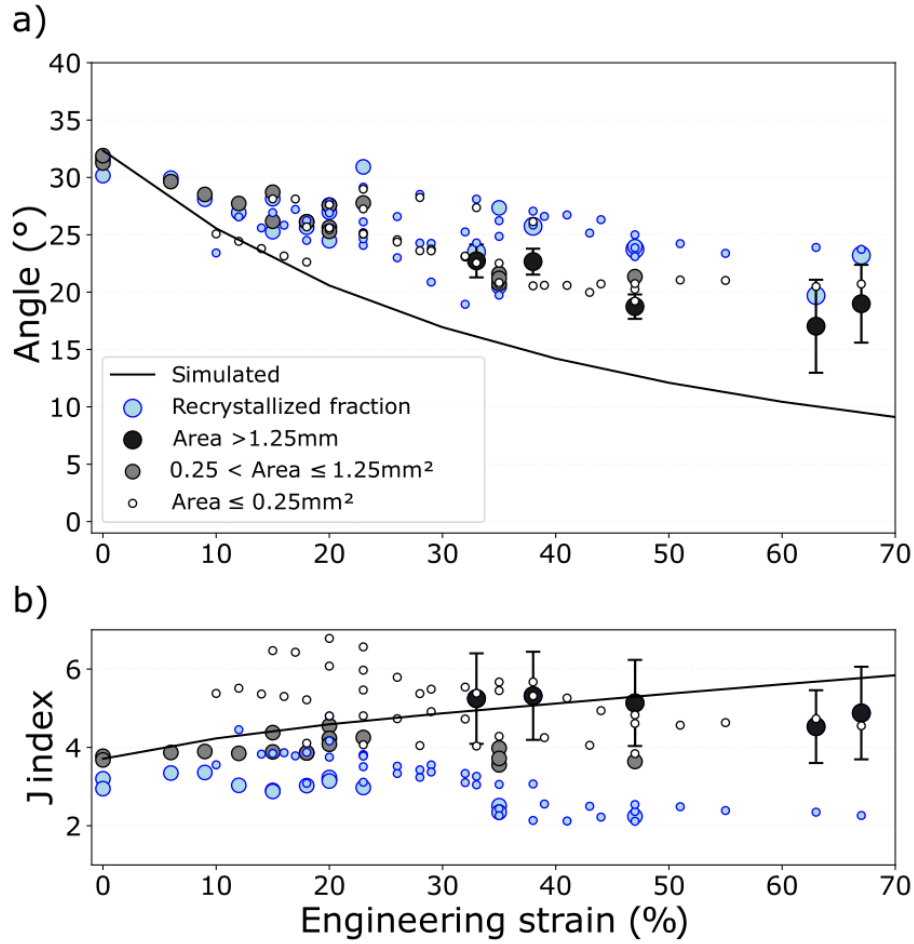


Figure 7: Evolution of the texture with engineering strain: (a) orientation of the texture, defined by the angle between the normal to the  $\langle c \rangle$  axis girdle and the traction direction and (b) texture strength, defined by the J-index. Symbols in white, gray, and black correspond to full orientation data in the map, considered as one orientation per pixel. Coarser black symbols mark data from larger, post-mortem maps. To estimate the spatial variability of the data and the representativity of the smaller in-situ maps, these large maps (area  $> 1.25 \text{ mm}^2$ ) have been re-sampled as multiple smaller maps with areas similar to that of the in-situ ones ( $\sim 0.2 \text{ mm}^2$ ) to obtain the mean and the standard deviation, represented by the errorbars, of the distributions. For comparison, the texture of the recrystallized fraction identified using a  $20 \mu\text{m}$  threshold in each map is represented by blue symbols; as for the full orientation data, the symbol sizes are proportional to the area of the map. The full lines indicate the texture evolution predicted by a 2<sup>nd</sup> order viscoplastic self-consistent simulation for uniaxial traction with normalized critical resolved shear stresses (CRSS) for the basal $\langle a \rangle$ , prismatic $\langle a \rangle$ , and pyramidal $\langle a+c \rangle$  systems of 1, 40, and 40 MPa and as the initial texture that of the starting material.

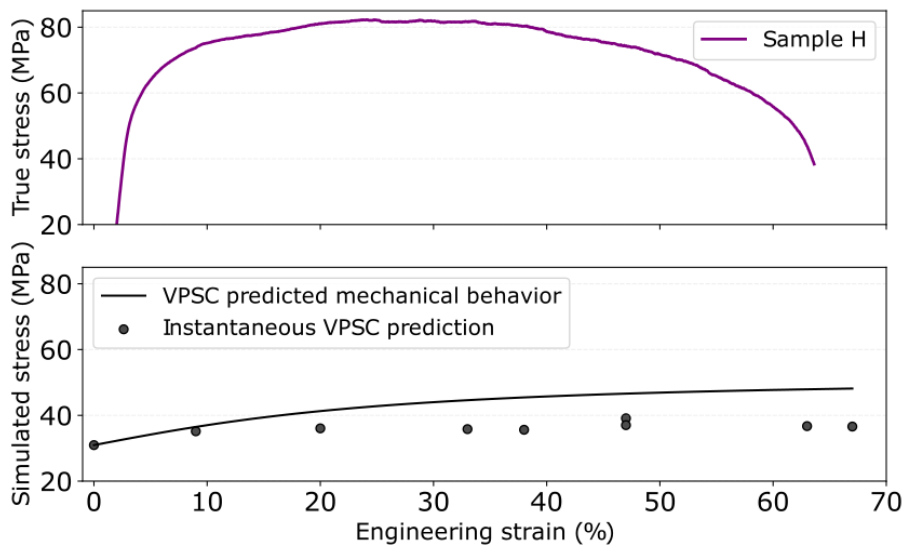


Figure 8: Comparison between the (a) mechanical behavior of Sample H, which was deformed without interruptions and (b) that predicted by a 2<sup>nd</sup> order viscoplastic self-consistent simulation for a monotonic uniaxial traction experiment with normalized critical resolved shear stresses (CRSS) for the basal<a>, prismatic<a>, and pyramidal<a+c> systems of 1, 40, and 40 MPa and the initial texture of sample Y and the instantaneous tensional stresses predicted using the same VPSC model for textures measured at different engineering strains.