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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} 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 μm
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} 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° 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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9 **3. Results**

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11 *3.1. Bulk mechanical behavior*

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

23
24 The behavior of Sample X is representative of the mechanical response of the
25 experiments in which the deformation was halted for in-situ EBSD mapping.
26 Each interruption induced a stress relaxation, which is manifested as a verti-
27 cal drop in the stress/strain curve. Three distinct deformation experiments
28 (0% to 35%; 35% to 47%; 47% to 67%) were performed sequentially on this
29 sample, with unloading and repolishing of the sample surface between the
30 experiments. The true stress/engineering strain curves of the three experi-
31 ments were assembled to construct the curve shown by the blue line in Fig.
32 1. The stress relaxation at 10% strain is a consequence of a reboot of the
33 tensile rig software during the experiment due to a technical issue.

34
35 The two strategies for EBSD acquisition presented in section 2.3 have been
36 tried on this sample: the segments of the curve displayed with full lines in
37 Fig. 1 present data for EBSD acquisition at 150°C, whereas those displayed as
38 dashed lines show the data for EBSD acquisition at 250°C. Despite the stress
39 relaxation due to the interruption of the traction during the EBSD acquisi-
40 tion, the mechanical behavior of the samples with in-situ EBSD acquisition
41 is consistent with that of sample H. It is characterized by elastic loading up
42 to 2% strain, transition from hardening to quasi-steady state around 10%
43 strain, and onset of softening around 35-37% strain. The stress peak at 35%
44 strain for sample X is a consequence of thermal contraction of the sample
45 as it was cooled down to room temperature. This might have caused some
46 hardening and explain the 10 MPa offset of the mechanical data for strains

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10 197 >35%. For the other experiments, unloading and cooling were synchronized
11 198 to avoid such stress peaks. Post-mortem optical analysis of the sample at
12 199 the end of the experiment highlights a stronger out-of-plane deformation and
13 200 incipient necking in a ~ 3 mm wide domain at the center of the specimen (see
14 201 Supplementary Material Fig. S3). In the experiments halted at lower strains,
15 202 necking is not observed.

17 203 *3.2. Bulk microstructural evolution*

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19 204 The increase in KAM values show a steady accumulation of lattice misorien-
20 205 tation (i.e., of GNDs) throughout the deformation experiment for all samples
21 206 (Fig. 2.a). The median increases quasi-linearly, but the KAM distribution
22 207 becomes progressive more skewed, due to faster increase of the higher values
23 208 relative to the median, marking the development of low angle grain bound-
24 209 aries with progressively higher misorientations. Domains characterized by
25 210 null KAMs are preserved over the entire deformation range. The data from
26 211 the first experiment on Sample X show slightly higher rates of lattice mis-
27 212 orientation accumulation than the remainder of the dataset. However, this
28 213 difference may result from a sampling bias, as these data have been collected
29 214 with a coarser step size than the remainder of the data (cf. Supplementary
30 215 Material Table T1).

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36 217 The grain size shows a two-step evolution (Fig. 2.b). All parameters de-
37 218 scribing the grain size distribution (median, first and third quartiles, and
38 219 maximum excluding outliers) decrease with strain up to 35% engineering
39 220 strain and remain almost constant afterwards. Median equivalent diameters
40 221 evolve abruptly from $>20 \mu\text{m}$ to $\sim 7 \mu\text{m}$ at strains $>35\%$. Note, however,
41 222 that this shift in behavior has not been observed within a continuous ex-
42 223 periment, but between successive experiments. Moreover, due to the limited
43 224 area of the domains analyzed during in-situ mapping, boundary grains could
44 225 not be eliminated from the dataset, biasing the estimation of the equivalent
45 226 diameters towards lower values.

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50 228 The RX fraction, that is, the area fraction of the map occupied by recryst-
51 229 tallized grains, defined using a 1° GOS threshold (see section 2.3) is initially
52 230 very high because of artifacts (Fig. 3.a). Indeed, undeformed samples have
53 231 “statically” recrystallized fractions ranging between 88-91%. As deforma-
54 232 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*

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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° ,
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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10 270 geometrically necessary dislocations is more pronounced than the changes in
11 271 grain boundary geometry. This observation indicates that, under the present
12 272 experimental conditions, dislocation mobility has faster kinetics than grain
13 273 boundary mobility.

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15 275 Analysis of the microstructure between 23% and 38% engineering strain for
16 276 Sample D documents continuation of the same trend (Fig. 5.a). The more
17 277 strained is the sample, the more serrated are the grain boundaries. The
18 278 progressive increase in the misorientation across subgrain boundaries is well
19 279 illustrated in the Mis2mean maps (right panel of Fig. 5.a). A zoom in the
20 280 abnormally coarse grain that occupies most of the lower part of the map
21 281 (Fig. 5.b) shows that the strong orientation gradient across the central part
22 282 of the grain is accommodated by a network of low-angle boundaries, 10-20
23 283 μm wide. This network became more complex and misoriented with strain
24 284 but did not evolve into a well-defined grain boundary. Most grains still have
25 285 sizes in the 30 to 100 μm range, but small DRX grains are observed in all
26 286 maps. The RX fraction based on GOS remains almost constant (Fig. 3.a),
27 287 but that based on grain size slowly increases (Fig. 3b). The discrepancy
28 288 between the two measures may be partially explained by the contribution of
29 289 polygonization and subgrain rotation to DRX. This process is well illustrated
30 290 by the DRX grain highlighted in green in Fig. 5.b, in which the new grain
31 291 boundaries, formed by polygonization, enclose preexisting substructure. This
32 292 process contrasts with nucleation by bulging, in which grain boundary mi-
33 293 gration cleans the dislocation substructure, producing DRX grains with little
34 294 internal misorientation.

35 295
36 296 A marked switch in mechanical behavior, from quasi steady-state to soften-
37 297 ing (Fig. 1), accompanied by stabilization of the previously decreasing grain
38 298 size (Fig. 2.b) and a sudden increase in the recrystallized fraction (Fig. 3)
39 299 is observed around 35% engineering strain. The evolution of the microstruc-
40 300 ture after this transition is well illustrated by data for sample X between
41 301 35% and 47% engineering strain (Fig. 6). In situ EBSD data for the 47% to
42 302 65% strain interval provide similar observations. The grain size distribution
43 303 is strongly bimodal with coarse grains (equivalent diameters $\geq 100 \mu\text{m}$) and
44 304 clusters of small DRX grains (equivalent diameters of 3-20 μm). Most DRX
45 305 grains show little intracrystalline misorientation (GOS $< 1^\circ$). In the 35% to
46 306 65% strain interval, the DRX clusters show fast microstructural evolutions
47 307 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° 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 2nd 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.

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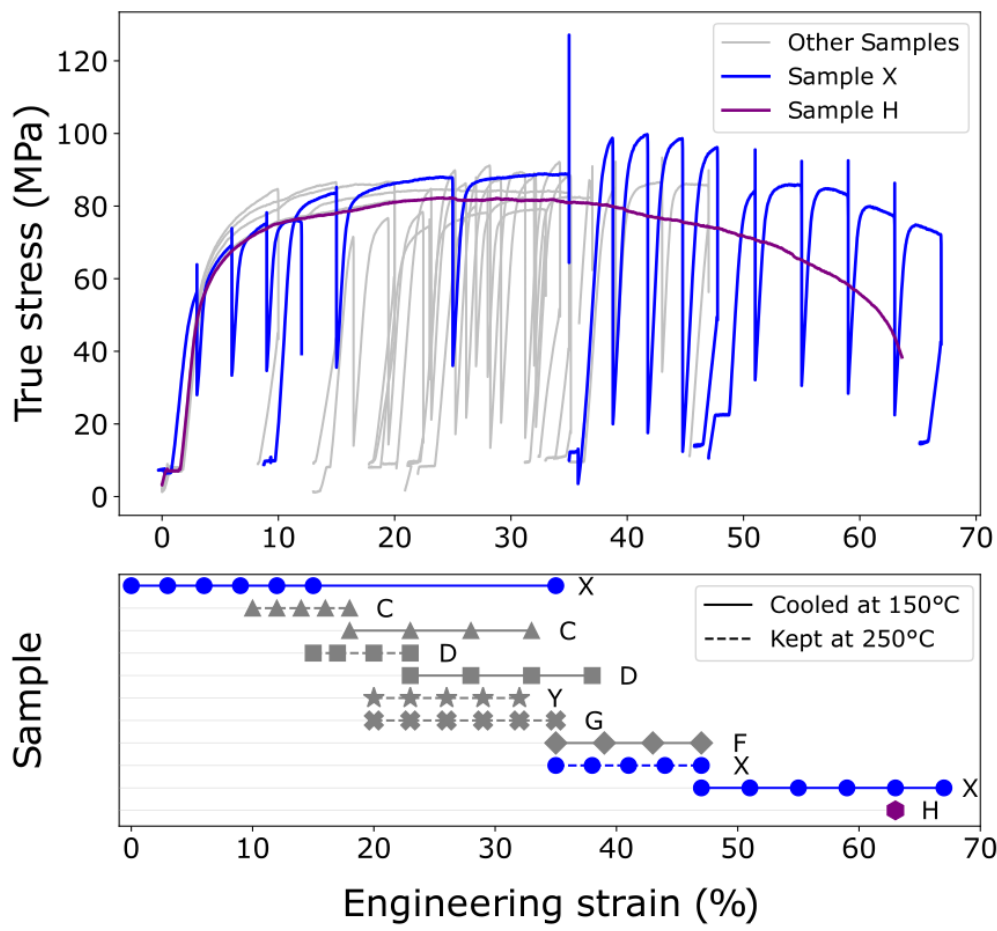


Figure 1: True stress/ engineering strain curves for all tests. Data for samples X and H, which cover the full strain range and are discussed in detail in the text, are highlighted in blue and purple, respectively. The lower panel details the experiments conducted on each sample: each line represents 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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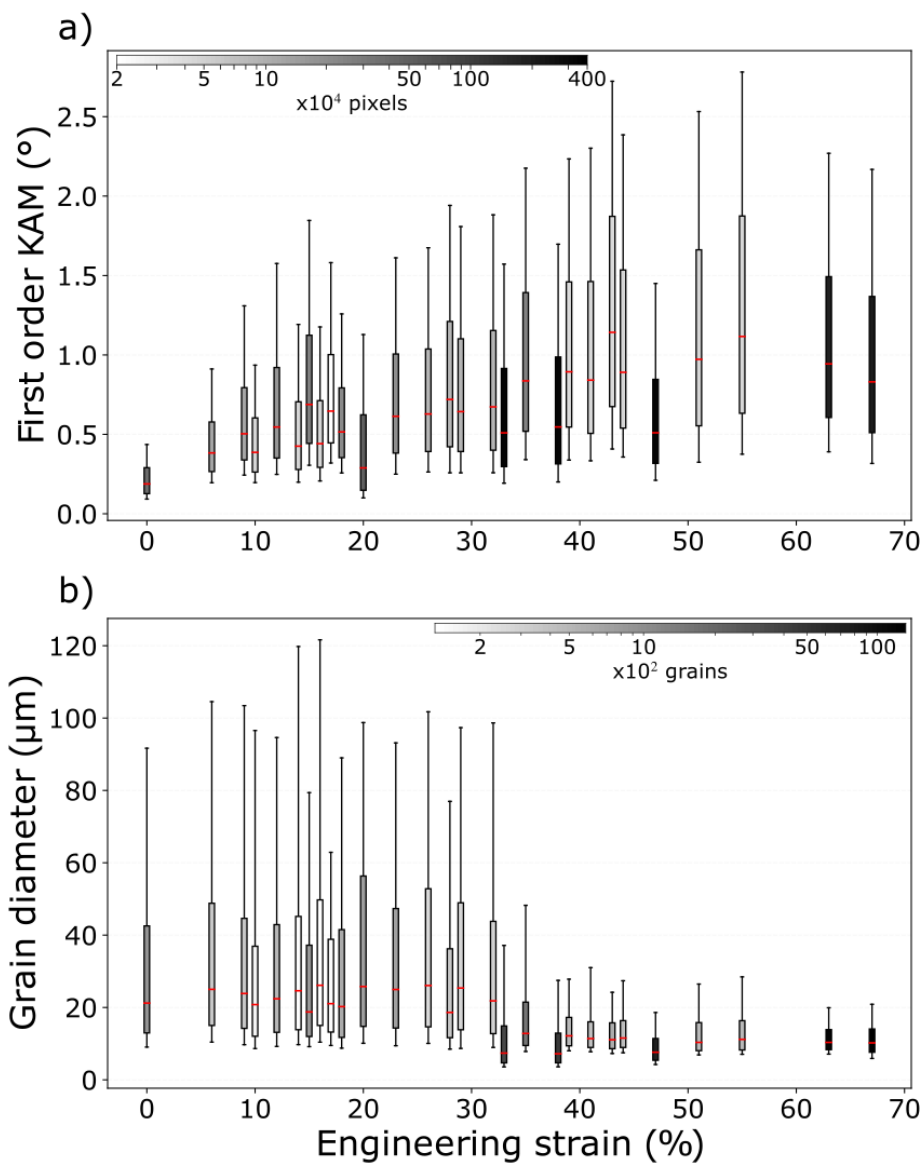


Figure 2: Evolution of the (a) intragranular misorientation and (b) grain size distributions with strain. Intragranular misorientation is defined by the 1st order Kernel Average Misorientation (KAM) and grain sizes, by the equivalent diameter. Data from multiple EBSD maps on a single or multiple samples 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).

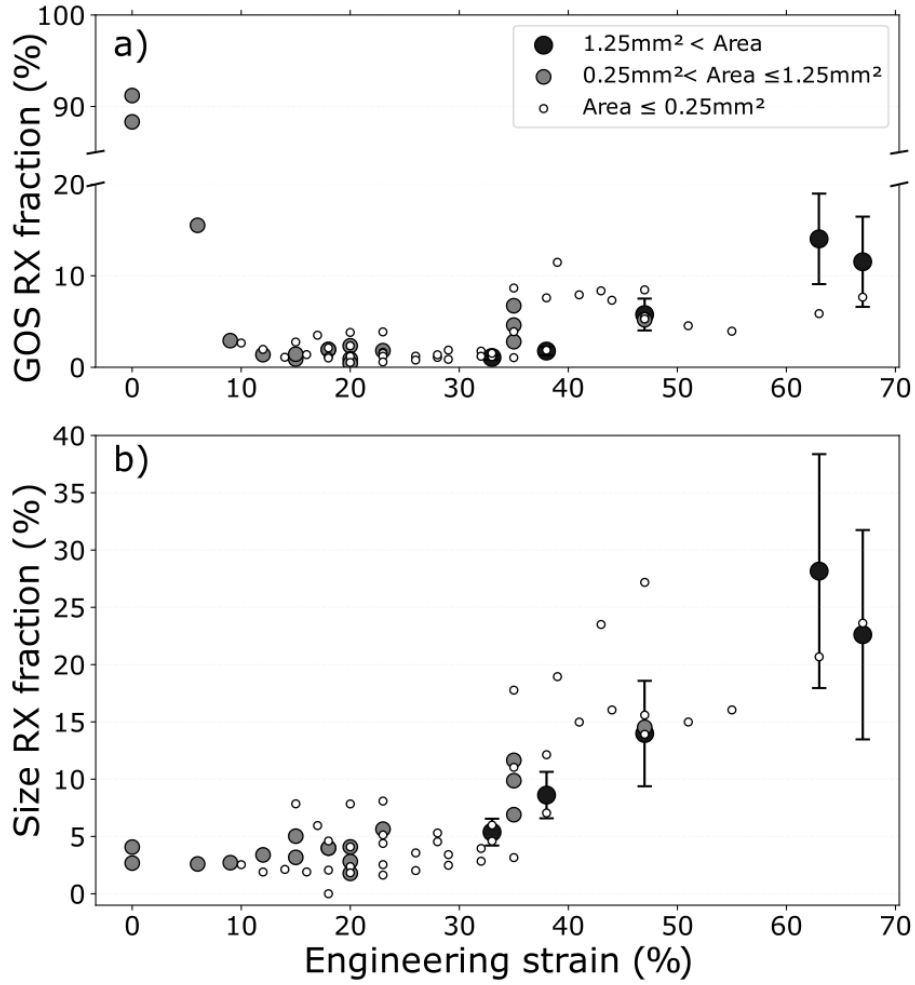


Figure 3: Evolution with strain of the recrystallized area fraction defined based on (a) a 1° GOS threshold and (b) a 20 μm equivalent diameter threshold. Symbol sizes indicate the total area of the EBSD map. To estimate the spatial variability of the data and the representativity of the smaller in-situ maps, the large maps (area > 1.25 mm²) have been re-sampled as multiple smaller maps with areas similar to that of the in-situ ones (~0,2 mm²); this allows to estimate the mean and standard deviation, represented by the errorbars, of the recrystallized area fraction distribution in these maps.

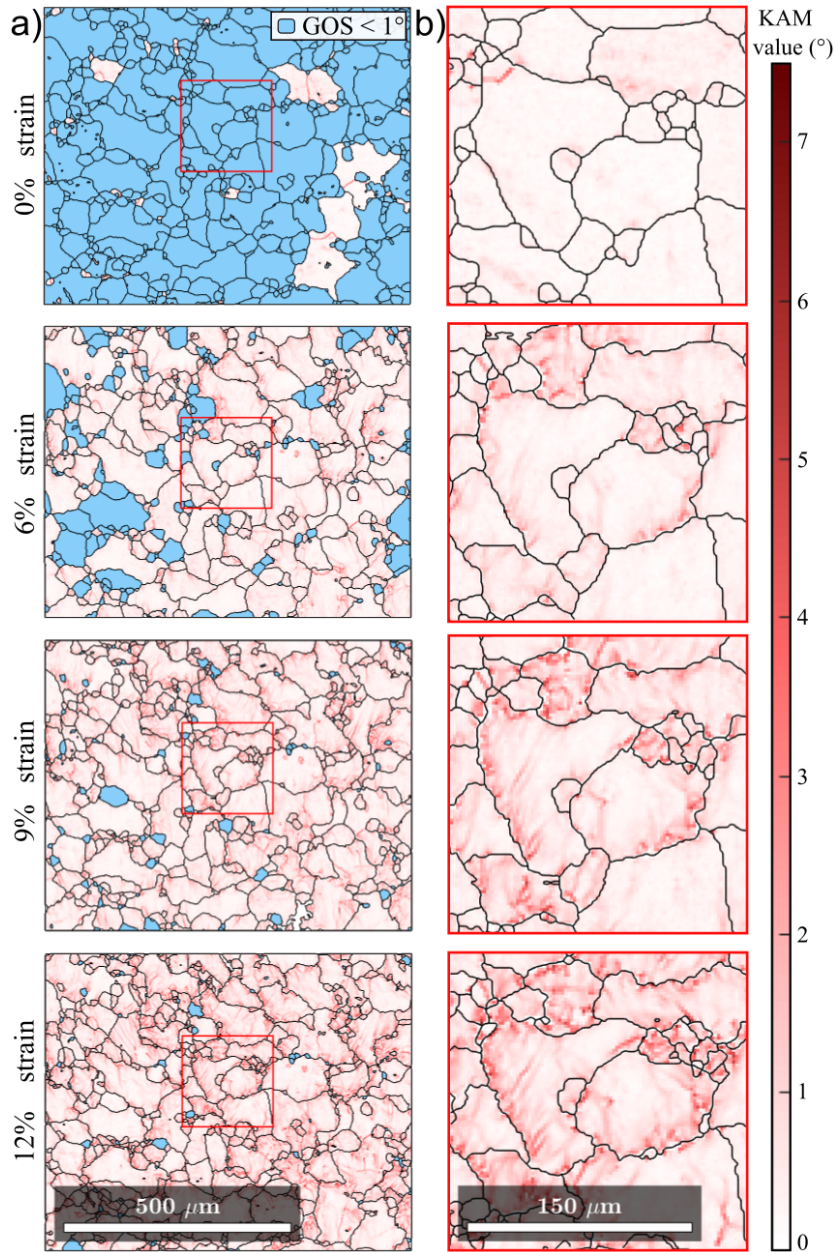


Figure 4: Microstructure evolution in the early stages of the experiments, illustrated by first order KAM maps obtained in situ at 0%, 6%, 9%, and 12% engineering strain for sample X. Grain boundaries, calculated using a 7.5° misorientation threshold, are represented as black lines. (a) Full size maps, in which the recrystallized grains identified using a 1° 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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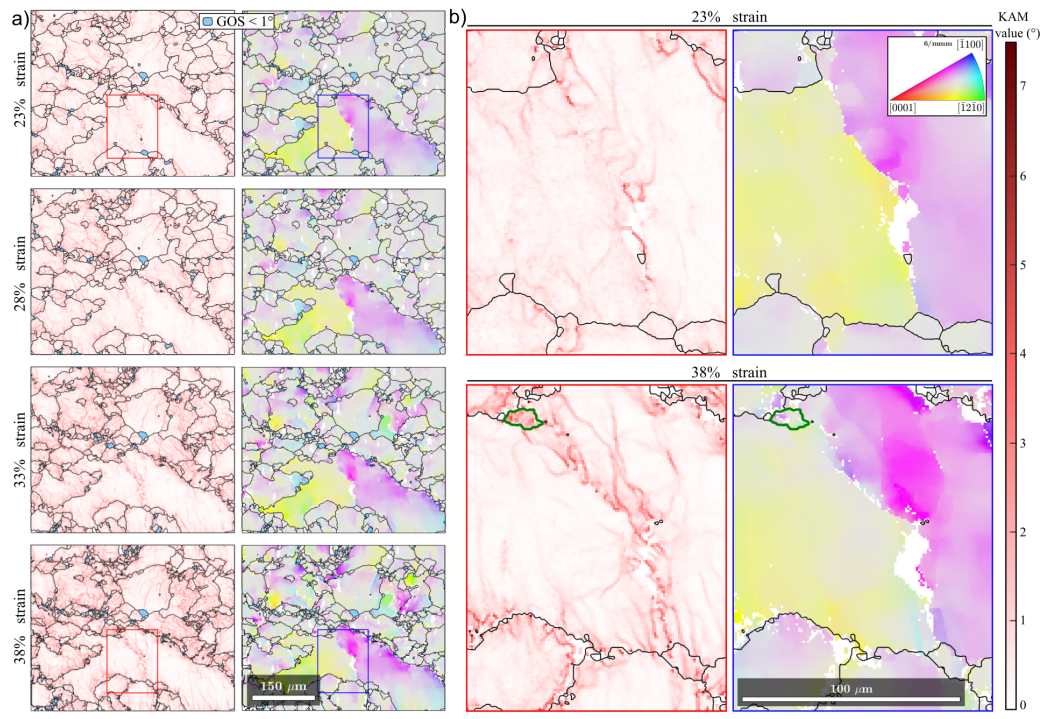


Figure 5: Microstructure evolution in the 23% to 38% strain interval, illustrated by in-situ first order KAM (left column) and mis2mean maps (right column) of sample D at 23%, 28%, 33%, and 38% engineering strain. Grain boundaries calculated using a 7.5° misorientation threshold are represented as black lines. (a) Full size maps, with recrystallized grains identified using a 1° 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.

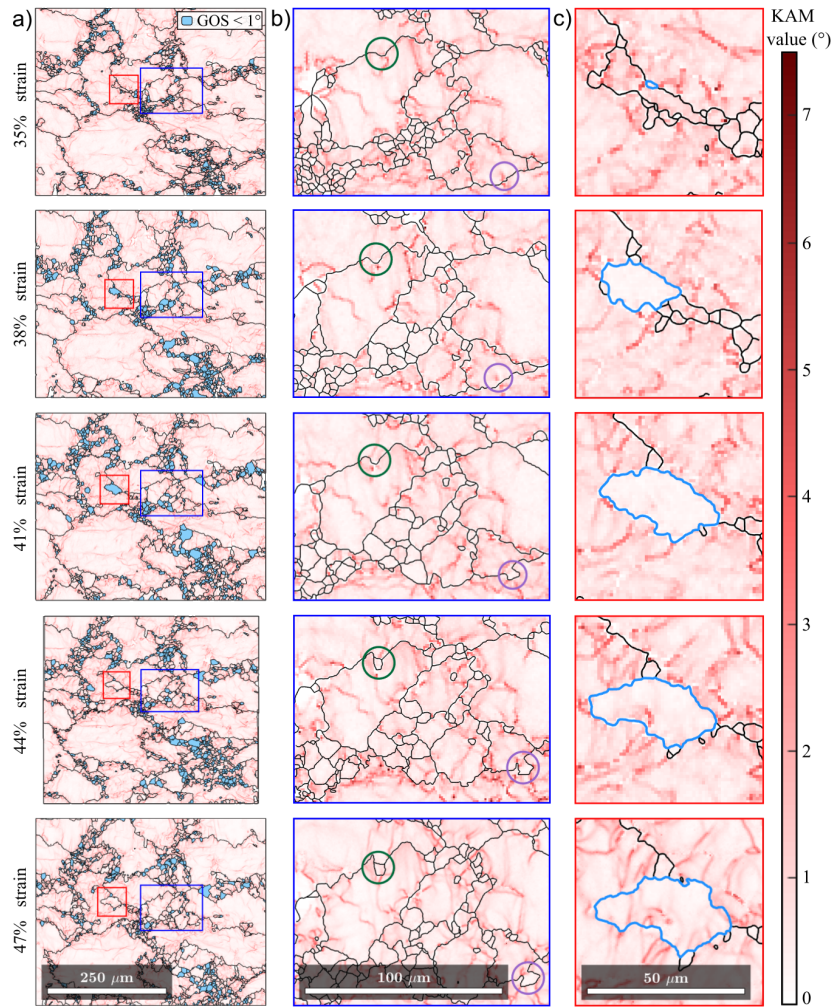


Figure 6: Microstructure evolution in the softening stage of the experiments, illustrated by KAM maps obtained in situ for sample X at 35%, 38%, 41%, 44%, and 47% engineering strain. All maps but that at 47% strain, were acquired with a $1 \mu\text{m}$ step size; the map at 47% strain was acquired with 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° misorientation threshold are represented by black lines. (a) Full size maps, in which recrystallized grains identified using a 1° 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.

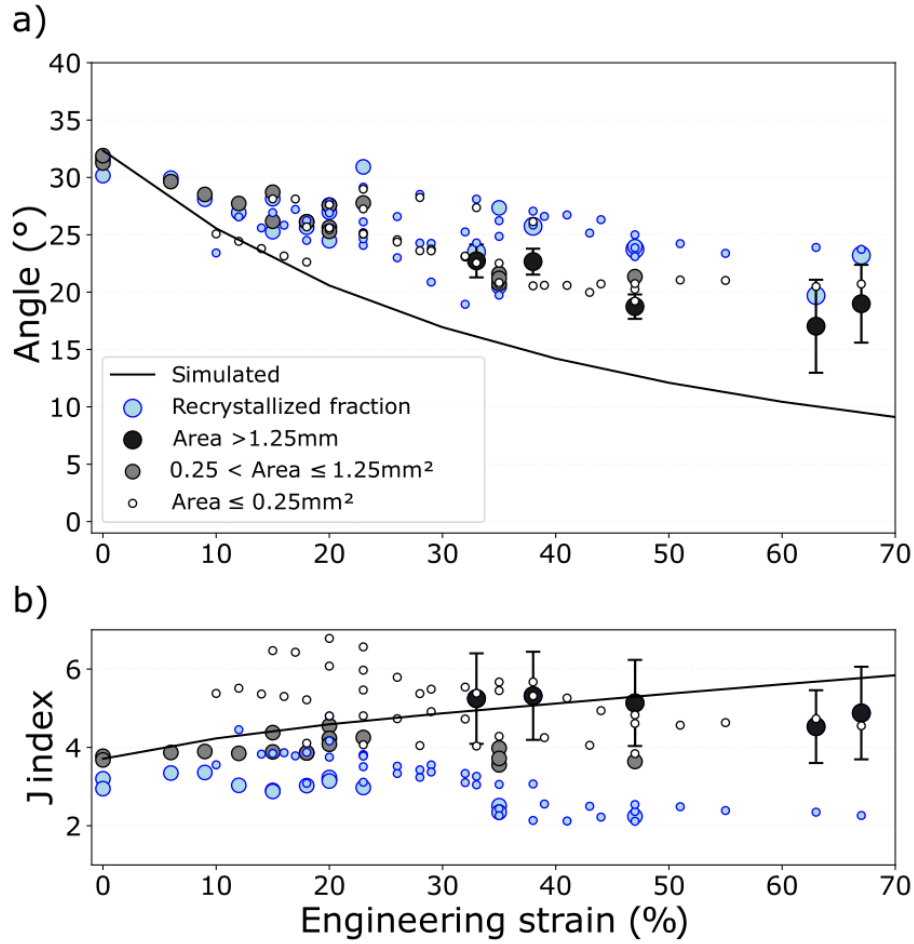


Figure 7: Evolution of the texture with engineering strain: (a) orientation of the texture, defined by the angle between the normal to the $\langle c \rangle$ axis girdle and the traction direction and (b) texture strength, defined by the J-index. Symbols in white, gray, and black correspond to full 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 2nd 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.

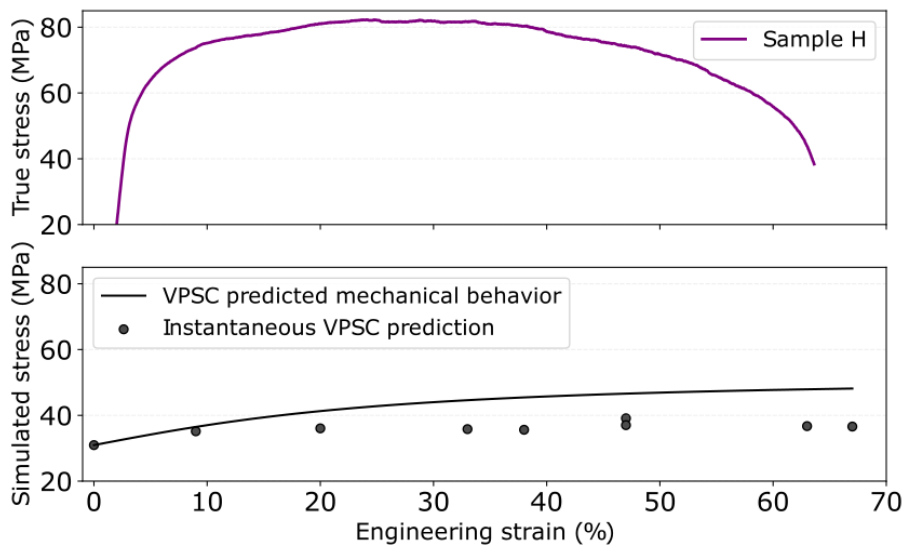


Figure 8: Comparison between the (a) mechanical behavior of Sample H, which was deformed without interruptions and (b) that predicted by a 2nd order viscoplastic self-consistent simulation for a monotonic uniaxial traction experiment with normalized critical resolved shear stresses (CRSS) for the basal<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.