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1 Creep Mechanisms in the Lithospheric Mantle Inferred from
2 Deformation of Iron-Free Forsterite Aggregates at 900-1200 °C

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16

17 **Abstract**

18 To further constrain the plasticity of rocks in the uppermost lithospheric mantle, deformation
19 experiments were carried out on forsterite aggregates using a gas-medium apparatus (Paterson press) at
20 300 MPa, 900-1200 °C and nearly constant strain rates of $\sim 10^{-5} \text{ s}^{-1}$. The starting material was a synthetic
21 iron-free forsterite aggregate with an average grain size of $\sim 2.8 \mu\text{m}$ and $\sim 2-3 \%$ of iron-free enstatite. Eight
22 deformation experiments were performed as well as an additional static annealing to characterize grain
23 growth. The maximum stresses obtained range from ~ 480 to 1870 MPa. Below 1000 °C, where stress
24 significantly exceeds confining pressure, and based on microstructural observations, grain boundary
25 mediated creep is observed, with evidences of sliding and cavitation (gaping) at grain boundaries. At 1050-
26 1200 °C, where pseudo-steady state could be achieved, the microstructures are very different and show
27 evidences of dislocation activity, resulting from the activation of several dislocation slip systems with
28 increasing temperature.

29 When compared to rheology laws previously obtained from similar experiments, the temperature
30 dependence of iron-free olivine creep is similar to the one of its iron-bearing counterpart at high temperature
31 ($\sim 1200 \text{ °C}$); at temperatures $\leq 1000 \text{ °C}$, however, the strength of iron-free olivine is higher than for iron-
32 bearing olivine. The deformation-induced textures obtained show that grain boundary sliding (GBS) lead to
33 cavitation, which was activated in response to large differential stresses, i.e., beyond the Goetze criterion.
34 Given these high-stress conditions, our results cannot be directly applied to deformation of the Earth's
35 mantle at large scale. Nevertheless, they highlight the key role played by grain boundaries in producing strain
36 at lithospheric temperatures, when crystal-plastic mechanisms remain inefficient.

37 **Keywords:** olivine, deformation, grain boundary sliding, upper mantle, high pressure

38 1. Introduction

39 The lithospheric mantle can be subject to temperatures as low as 500 °C. Experimental
40 deformation of mantle rocks at such low temperatures is a major challenge in mineral and rock
41 physics, since the strain rates necessary to achieve steady state dislocation creep are too low to be
42 performed in the laboratory. Consequently, deformation experiments that provide insights into the
43 “cold” rheology of olivine, are performed at high stresses without achieving geologically relevant
44 steady state and the strain rate dependence is difficult to assess (Druiventak et al., 2011; Mei et al.,
45 2010). Typically, little to no rate-dependence is observed because it is impossible to activate ductile
46 flow, which leads to seemingly large stress exponent values in the flow law (Druiventak et al., 2011).
47 For decades, the flow law of Evans and Goetze (1979), which uses an exponential expression to
48 account for the Peierls stress control over dislocation motion, has been used to model the low-
49 temperature rheology of the olivine-rich rocks composing the uppermost mantle. Nevertheless,
50 recent experimental studies have shown that the uppermost mantle is likely much weaker than
51 previously expected from extrapolations of high-temperature rheology flow laws to upper mantle
52 temperatures (e.g., Demouchy et al., 2009, 2013, 2014, Boioli et al., 2015a, 2015b; Thieme et al.,
53 2018; Gouriet et al., 2019).

54 A solution to these issues, is to identify the physical deformation mechanisms at play (e.g.,
55 dislocation slip systems) and to implement them in numerical dislocation dynamics models (Boioli
56 et al., 2015a, 2015b, Gouriet et al., 2019). Although this approach gives valuable insights into single
57 crystal plasticity, it does not fully describe the creep behavior of polycrystalline rocks, which may
58 arise from a variety of interacting deformation mechanisms. Therefore, to grasp the complexity
59 and better constrain the low-temperature rheology of mantle rocks, and despite the issues
60 mentioned above, experiments on olivine aggregates are still needed.

61 The incorporation of iron, hydrogen and titanium in the atomic structure of olivine is known
62 to induce a weakening effect (iron: e.g., Zhao et al., 2009, 2018; Bollinger et al., 2015; Hansen et

63 al., 2012; hydrogen: e.g., Mackwell et al., 1985; Mei and Kohlstedt, 2000; Tasaka et al., 2016; Tielke
64 et al., 2017; titanium: e.g., Faul et al., 2016; Cline II et al., 2018). To avoid adding complexity to the
65 olivine plasticity due to its dependence upon point defects (i.e., vacancy concentrations), it was thus
66 chosen here to restrict the deformation experiments to hydrogen-free, iron-free fine-grained
67 forsterite-rich aggregates.

68 We used a two-phase aggregate material, mostly composed of forsterite (with minor amounts
69 of enstatite), which has previously been used in several deformation studies at low pressure and
70 high temperature, in the diffusion creep regime (Tasaka et al., 2013; Nakakoji et al., 2018). The aim
71 of the present study is to assess the creep mechanisms of this fully equilibrated fine-grained
72 aggregate under conditions more relevant to the uppermost mantle, i.e., under significant confining
73 pressure and colder temperatures. In particular, we focus here on a temperature range that
74 encompasses the transition from power law flow (high temperature, $T > 0.6 T_m$, where T_m is
75 melting temperature; for Fo_{100} , $T_m=1890$ °C) to exponential law flow (“cold” temperature, 0.3-
76 $0.6 T_m$). In addition to the mechanical data, the microstructures are analyzed with state-of-the-art
77 techniques to provide valuable information regarding deformation mechanisms.

78

79 **2. Materials and Methods**

80 **2.1 Starting material**

81 The samples used in this experimental study were produced from synthetic nano-sized
82 powders at the Earthquake Research Institute (Tokyo, Japan), following a sintering procedure
83 previously described in details by Koizumi et al. (2010), which we only briefly outline here. Starting
84 nano-sized powders of forsterite and enstatite were first synthesized from high purity SiO_2 and
85 $Mg(OH)_2$, then pre-pressed into the desired cylindrical shape in carbide dies, and pressed under
86 hydrostatic conditions at 200 MPa. The resulting cold-pressed specimens were then annealed under

87 vacuum at 1260 °C for 2-3 hours, which yields a fully dense aggregate (< 1 % porosity) and ensures
88 complete dehydration (Koizumi et al., 2010). The chemical composition of the starting material
89 was analyzed by X-ray fluorescence with a Philips® PW 2400 spectrometer (see Supp. Mat. Table
90 S1). Iron content is under 10 ppm. This new type of high-quality sintered aggregate has proven to
91 be an excellent material for rheology studies (Hiraga et al., 2013; Tasaka et al., 2013). The
92 microstructure of the starting material was characterized by scanning electron microscopy (SEM)
93 and electron backscatter diffraction (EBSD). The EBSD map in Figure 1 shows that the bi-
94 minerallic aggregate is composed of pure forsterite with an average equivalent diameter of 2.8 μm.
95 It also contains ~2-3 % of orthoenstatite homogeneously distributed across the aggregate with a
96 smaller grain size than forsterite (1.8 μm on average), which prevents it from impacting the bulk
97 rheological properties (Huet et al., 2014; Ji et al. 2001). The EBSD maps also provide statistical
98 data on the microstructure, as reported in Table 1. The starting material shows equilibrated textures,
99 with ubiquitous triple junctions and straight grain boundaries (Figure 1), as already described by
100 Koizumi et al. (2010). Due to the very fine grain size, initial porosity is difficult to assess from SEM
101 images since it is difficult to differentiate between actual residual pores from sample sintering and
102 newly formed cavities caused by grain plucking during cross section preparation. Nevertheless,
103 porosity has been characterized in previous studies and is estimated to be significantly less than
104 1 % in volume (Koizumi et al., 2010). As observed on Figure 1, the grains are nearly equiaxed.
105 Their aspect ratio is found here to be 1.9, whereas previous studies using the same material have
106 reported a value of 1.3-1.4 (Koizumi et al., 2010). The aggregate starting material has a measurable
107 shape preferred orientation (SPO), most likely inherited from the cold press stage of the samples
108 synthesis (Koizumi et al., 2010). The samples used for deformation consisted in cylinders with
109 diameters ranging from 4.5 to 4.8 mm, and lengths varying between 7.25 and 10.55 mm (Table 1).

110

111 2.2. Deformation experiments

112 Eight deformation experiments were carried out using a high-resolution gas-medium high-
113 pressure high-temperature apparatus (Demouchy et al., 2013, 2014; Paterson, 1990, Thieme et al.,
114 2018) at Geosciences Montpellier (University of Montpellier, France). An additional static
115 annealing test was carried out without deformation to assess the importance of grain growth during
116 our experiments (NF_1000-0, Table 1). All experiments were performed under a confining
117 pressure of 300 MPa using argon gas. Constant piston-displacement uniaxial deformation was
118 performed under constant temperatures ranging from 900 to 1200 °C (Table 1). The samples were
119 placed between alumina and zirconia pistons and fitted in metal sleeves (e.g., Mei and Kohlstedt,
120 2000; Demouchy et al., 2014). The nature of the metal sleeve was chosen depending on the
121 temperature of each experiment, to avoid melting of the sleeve while keeping its strength as low as
122 possible (Table 1). Silver, copper and nickel sleeves were used for experiments at <1000 °C, 1000-
123 1050 °C, and >1050 °C, respectively (Table 1). All parts were encapsulated in an iron jacket thin
124 enough to transmit the confining pressure.

125 Axial force values were obtained from an internal load cell during deformation. Flow laws for
126 Cu, Fe, Ni, and Ag from Frost and Ashby (1982) were used to determine the contributions of the
127 iron jacket and the metal sleeve to the measured load. Sample stress as a function of time was then
128 retrieved by calculating the evolution of the sample surface corresponding to a shortening with
129 constant volume. Strain was corrected using the apparatus stiffness, which was calibrated
130 beforehand ($\sim 83 \text{ kN mm}^{-1}$). The stress-strain data were further processed (smoothed) to accurately
131 identify the yield point and the apparent Young moduli at yield point (see Supp. Mat. for details,
132 Figures S1 and S2).

133 For each experiment, constant displacement rate was set based on the initial length of the
134 sample to yield a stress plateau with a strain rate of 10^{-5} s^{-1} . All samples were deformed, at least,
135 until the maximum stress was achieved. Below 1050 °C, axial shortening was stopped if a significant
136 stress decrease—indicative of brittle failure of the sample—was observed. At temperatures

137 > 1050 °C, a plateau with constant stress was obtained and more extensive deformation was
138 performed. Finite strain ranged from 2.8 to 12.4 % (Table 1).

139

140 **2.3 Scanning electron microscopy and electron backscattered diffraction**

141 The recovered samples were kept in the metal sleeve and jacket and cross sections were cut
142 either normal or parallel to the compression direction for SEM and EBSD. The sample sections
143 were prepared using a standardized polishing protocol (Thieme et al., 2018). A final chemo-
144 mechanical polishing step using colloidal silica on a vibrating plate was necessary to achieve a high-
145 quality polish for EBSD. The SEM and EBSD analyses were performed with a CamScan X500FE
146 Crystal Probe equipped with an EBSD system at Geosciences Montpellier (University of
147 Montpellier, France). The geometry of the Crystal Probe-EBSD was previously detailed in
148 Demouchy et al. (2011). Operating conditions were 15-18 kV, ~6 or 10 nA (for exposure times of
149 48 and 24 ms, respectively) and a working distance of 24-25 mm. Low vacuum conditions (4 Pa of
150 gaseous nitrogen), carbon coating and copper-carbon tape were used around the studied area to
151 avoid charging of the specimen. Several EBSD maps were collected on each sample section,
152 including on the starting material (undeformed), as well as on the statically annealed sample.

153 The dimensions of the area mapped by EBSD typically varied from a few tens to several
154 hundreds of microns, which corresponds to hundreds to several thousands of grains, thus allowing
155 satisfying statistical analysis in terms of grain size, shape grain and crystallographic orientation. The
156 step size was set either to 0.2 or 0.4 μm depending on the size of the analyzed area.

157 The data were acquired using Oxford instrument's HKL Aztec2 software. Data were first
158 processed to remove wild spikes (isolated pixels that have eight similar neighbors) and fill non-
159 indexed pixels that had seven neighbors with identical orientations, then treated with the MTEX
160 MATLAB toolbox (Bachmann et al., 2010; Hielscher and Schaeben, 2008; Mainprice et al., 2015).

161 Grain boundaries are usually detected using a critical misorientation angle of 10-15° (Mainprice et
162 al., 2015). Here, grain boundaries were detected using a threshold misorientation angle of 13°.
163 Variations of this critical threshold angle between 10 and 15° does not impact the microstructure
164 results significantly (see Supp. Mat., Table S2). Grains were filtered to a minimum of 1.44 μm^2 (i.e.,
165 9 or 36 pixels, according to the step size). Similarly, subgrains were identified as neighboring pixels
166 with misorientations of 2-13°. The density of the orientation distribution function was calculated
167 using an axially symmetric de la Vallee Poussin kernel, with a half-width of 10° (bandwidth of 28
168 in spherical harmonic coefficients). Crystal preferred orientations (CPO) and texture J-index were
169 calculated (Bunge, 1982). The texture J-index is a measurement of the texture strength calculated
170 as the integral of the square of the orientation distribution function, and is calculated using one
171 data point per grain to avoid overestimating the contribution of large grains (Mainprice et al., 2015).
172 Densities of pole figures were normalized to a uniform distribution and contoured at intervals of
173 $0.06 \times \text{uniform}$.

174

175 **2.4 Transmission electron microscopy**

176 Transmission electron microscopy (TEM) was also used to characterize the microstructures
177 after the deformation experiments. Three deformed samples (NF_950-1, NF_1050-1 and
178 NF_1200-1) were selected for this investigation from which doubly polished thin sections (30 μm
179 thick) were prepared. Sample NF_1200-1 was cut parallel to the deformation axis, and NF_950-1
180 and NF_1050-1 perpendicular to the deformation axis. They were glued on a Cu grid and ion milled
181 at 5 kV under a low beam angle of 15° until electron transparency was reached. The foils were
182 subsequently covered with a thin layer of carbon. TEM observations were carried out at the
183 University of Lille (France) using a FEI® Tecnai G2-20 TWIN microscope operating at 200 kV.

184

185 3. Results

186 3.1 Mechanical data

187 The stress-strain curves from each deformation experiment are shown in Figure 2. Stress data
188 for experiment NF_1110-1 appear noisier than other experiments due to a temporary grounding
189 issue of the internal load cell during the deformation, which resulted in apparent stress fluctuations
190 (Figure 2a). Maximum stresses range from 480 to 1870 MPa, for temperatures varying from 1200
191 to 900 °C, respectively. The mechanical values reported in Table 1 thus define an expected inverse
192 dependence with increasing temperature. At temperatures ≤ 1000 °C, stress increases until it
193 reaches a maximum, then starts decreasing upon further straining. Sample failure was observed for
194 NF_900-1. For experiments NF_950-1, NF_1000-1 and NF_1000-2, deformation was stopped
195 after observing a significant stress decrease, indicative of possible imminent failure. In contrast, at
196 higher temperatures (> 1000 °C), stress reaches a constant value (plateau) and samples could be
197 further deformed in an apparent (quasi-) steady state. We note however, that, for all experiments,
198 the stress increases above the confining pressure (> 300 MPa) and is upheld without immediate
199 failure over non-negligible amounts of strain, i.e., several percent after apparatus stiffness
200 correction.

201 From 900 to 1110 °C, yield stress values are close to ~ 600 MPa, regardless of temperature.
202 Yield stress is only significantly lower in experiment NF1200-1, performed at the highest
203 temperature (Figure 2b). In contrast, aside from experiment NF1110-1, we observe a consistent
204 decrease in the apparent Young modulus with temperature, from 112 GPa at 900 °C to 32 GPa at
205 1200 °C (Table 1).

206 3.2 Microstructures

207 Results from EBSD analyses on the deformed aggregates are summarized in Table 1. Average
208 grain size, aspect ratio and shape factor (i.e., measured perimeter/perimeter of the circle with

209 equivalent area) values obtained for the largest map in each sample, are shown as a function of
210 finite strain and temperature in Figure 3. For the sake of readability, a full statistical report is
211 provided in the supplementary material, where results from the two largest maps for each sample
212 are given along with corresponding minimum, maximum and standard deviation values (Table S3).
213 We note that values of aspect ratio are somewhat larger than expected. This is attributed to a slight
214 drift of the electron beam (often observed when working with non-conductive samples) when
215 collecting EBSD maps, which results in a minor artificial elongation of the grains.

216 From the EBSD maps, average grain sizes of 2.7-3.3 μm are found. Therefore, both in the
217 static annealing test NF_1000-0 and in the deformation experiments, grain size remains essentially
218 unchanged from the value measured in the starting material. Only a slight decrease (-15%) in grain
219 size is observed in the deformed samples (Table 1, Figure 3). Nevertheless, one finds no correlation
220 between grain size and temperature or strain over the entire data set (Figure 3), consistently with
221 the results of Tasaka et al. (2013), who reported only minor grain growth, even at temperatures as
222 high as 1260 $^{\circ}\text{C}$. Based on the low temperatures and short durations of the present experiments,
223 static growth is therefore negligible here (Table 1, Figure 3).

224 SEM images displayed in Figure 4, show the preservation of equilibrium texture features, such
225 as triple junctions and straight boundaries in the samples deformed at 950-1000 $^{\circ}\text{C}$. Visually,
226 textures from samples deformed more extensively and at greater temperatures (1050-1200 $^{\circ}\text{C}$)
227 show more curved grain boundaries (Figure 4g, h). This is confirmed by the values of shape factor
228 and aspect ratio, which remain essentially unchanged, when compared to the starting material, for
229 the samples deformed at 900-1000 $^{\circ}\text{C}$, as well as for NF1110-1 (all of which experienced less than
230 10 % strain), but are slightly larger for the three samples deformed by at least 10 % strain at 1050-
231 1200 $^{\circ}\text{C}$ (Table 1, Figure 3). In the low-temperature samples, many grain boundaries are open
232 (Figure 4), a feature that seems much less common at 1000-1110 $^{\circ}\text{C}$ and which is not observed at
233 1200 $^{\circ}\text{C}$.

234 In addition, at low temperature, many grains appear with strong topology in the SEM images
235 (Figure 4b), which is due to out-of-plane motion (vertical offsets) between adjacent grains in the
236 imaged plane. At 1050 °C, voids created by the opening of grain boundaries are still observed.
237 Typical cases where grains have slid relative to each other and created a void due to insufficient
238 ductility of adjacent grains can be seen in Figure 4d.

239 In all samples deformed at 1050 °C and 1100 °C, grain boundaries are serrated in a pervasive
240 way as shown in Figure 4. At 1050 °C, the scale of the serration (its wavelength) is at the limit of
241 the observation capability of the SEM, i.e., in the order of tens of nanometers, at most. We note
242 that, due to the relatively small scale at which this change occurs, it is not measurable in terms of
243 shape factor (which increases with grain boundary tortuosity, Figure 3) since EBSD data are
244 collected with much larger step sizes than the serration scale. Nevertheless, the wavelength of the
245 serration seems to increase with temperature and decreasing stress; as also suggested by TEM
246 images of samples NF_950-1 and NF_1050-1 (see below for details). At 1110 °C, the samples show
247 no evidence of major grain boundary sliding but the serration of grain boundaries is ubiquitous and
248 occurs at the scale of a few tens to hundreds of nanometers. Several large grains exhibit important
249 intracrystalline contrast variations, typical of subgrain boundaries (Figure 4f). This results from
250 distortions of the crystal lattice and/or from defect concentrations and therefore highlights active
251 intracrystalline plastic deformation. At 1200 °C, all grain boundaries remained closed and many
252 display incipient bulging, with wavy boundaries at the scale of hundreds of nanometers to a micron
253 (Figure 4h). Neither gaping, nor grain boundary sliding is observed.

254 The distribution of the long-axis direction of the grains is shown in Figure 5. A strengthening
255 of the initial SPO (i.e., present in the undeformed sample) is observed upon deformation (Figure
256 5 and Table S3). Indeed, the long-axis of the grains tend to align normal to the compression axis,
257 which is the same as during the compaction stage of the samples, prior to sintering. We note that
258 for samples NF_1110-1 and NF_1110-2, shortened by 12.4 and 5 %, respectively, the long axis

259 orientation distribution is very similar, which implies that most of the SPO formed during the early
260 stages of the axial deformation.

261 By definition, grain orientation spread (GOS) defines the variations in local crystallographic
262 orientation inside a given grain. Therefore, in an ideal unstrained crystal lattice, GOS is theoretically
263 nil and its actual value only reflects EBSD measurement uncertainty. This is the case for the
264 undeformed samples, which have an average GOS of 0.2-0.21° (Table 1 and Figure 3). Larger GOS
265 values can reflect plastic deformation due to the presence of internal strain heterogeneities of the
266 crystal lattice, which can originate from residual elastic strains (although unlikely here, in the case
267 of a free surface) or more generally from the presence of geometrically necessary dislocations
268 (GND). In the EBSD maps, GOS increases with the amount of strain to reach a maximum of
269 1.375° at 1100 °C. The largest values are obtained for samples NF_1110-1 and NF_1050-1
270 (Figure 3), which reveal substantial intracrystalline plasticity. At higher temperature, increasing
271 temperature seems to have the opposite effect since lower values of GOS (0.705°) were obtained
272 for sample NF_1200-1, deformed by about the same amount (Table 1). A comparison between
273 two EBSD maps collected on samples that have experienced different temperatures and finite
274 strains is shown in Figure 6. Similar to undulose extinction in optical microscopy, Misorientation-
275 to-Mean (Mis2Mean), which represents angular variations in crystal orientation inside a grain
276 relative to the grain's average crystallographic orientation, allows the spatial strain heterogeneity
277 distributions of the crystal lattice to be visualized. When finite strain is the largest, Mis2Mean values,
278 and therefore GOS, often exceed 5°, as evidenced by the brighter areas in Figure 6b, Mis2Mean
279 values > 5° are more common in coarser grains, where subgrains often formed. We note that, in
280 Figure 6a, yellow-saturated grains are a consequence of neighboring grains being incidentally
281 positioned in crystallographically close orientations and therefore identified as single grains with
282 large Mis2Mean values, whereas the actual maximum value is ~1°.

283 Pole figures obtained for the starting material, the annealing test and the deformation
284 experiments are displayed in Figure 7 and show near-random crystal orientations, as pointed out
285 by their very low J -indexes ranging from ~ 1.01 - 1.11 . However, in many cases, analysis over a large
286 number of grains ($> 10,000$) detected slight but meaningful deviations from a random distribution.
287 As expected, the starting material shows no significant CPO. Samples deformed from 900 to
288 1000 °C experienced small amounts of finite strain and do not show any significant CPO either.
289 Only sample NF_950-1 displays a possibly meaningful pattern, where the [001] axis forms a girdle
290 normal to the compression axis. This very weak CPO pattern may also be present in other samples
291 but was probably only detected in NF_950-1 due to the large EBSD map size, which allowed better
292 statistics (over 14,000 grains analyzed). We note, indeed, that J -indexes are consistently higher for
293 smaller EBSD maps, which is an artifact of poorer statistics generating greater local maxima. In
294 the present case, the very weak texture described above was likely present in the sample prior to
295 deformation and preserved due to very low finite strain.

296 In contrast, three out of four samples deformed between 1050 and 1200 °C (≥ 10 % strain),
297 NF_1050-1, NF_1110-1 and NF_1200-1, show a weak CPO characterized by an alignment of the
298 [010] axis parallel to the compression direction. The distribution of [001] axes defines a girdle
299 normal to the shortening direction in NF_1050-1, whereas it displays maxima at $\sim 45^\circ$ of the
300 shortening direction in NF_1110-1, and is nearly random for NF_1200-1. Similarly, [100] axes have
301 local maxima at 45° of the compression direction in NF_1050-1 and NF_1110-1. We note that these
302 features appear fainter for sample NF_1200-1. In addition, this CPO is not observed for sample
303 NF_1110-2, which, consistently with its low finite strain, shows a texture similar to that of the
304 starting material and the low temperature samples.

305 In summary, deformation-induced CPOs are characterized by a concentration of [010] axes
306 parallel to the compression direction, while the other axes tend to form girdles and point maxima,
307 which are normal and at $\sim 45^\circ$ with respect to the compression direction, respectively. These

308 patterns are characteristics of the A-fiber texture, where the [100] and [010] axes are parallel to the
309 shearing (lineation) and principal stress directions, respectively (Mainprice et al., 2005; Michibayashi
310 et al., 2016; Ohuchi et al., 2015; Tommasi et al., 2000). Similar textures have also been reported for
311 iron-bearing olivine using the same experimental setup at 900 °C and are indicative of co-existing
312 [100](010), [001](010) and possibly [001]{110} slip systems (Demouchy et al., 2009; Demouchy et
313 al., 2014; Phakey et al., 1972; Raleigh, 1968).

314 Intracrystalline plasticity was identified in the EBSD maps of the deformed samples via the
315 analysis of misorientation within the grains determined by MTEX. At 1050-1200 °C, many
316 subgrains were observed (misorientations of 2-13°) and statistics on those subgrains could be
317 extracted (Figure 8, Figures S3 and S4). At 900-1000 °C, however, little intragranular plasticity and
318 few subgrains are present. Intracrystalline plasticity was therefore investigated by analyzing targeted
319 grains that showed a GOS > 1° and where deformed areas larger than 10 pixels could be analyzed
320 (Figure 8 and Figure S5). Subgrains can form twist or tilt walls in the grains. Figure 8 (and Supp.
321 Mat. Figures S3 and S4) illustrates the types of subgrains identified in all three samples that have
322 experienced major deformation; i.e., NF_1050-1, NF_1110-1 and NF_1200-1. Subgrains are
323 mostly found in large grains (>~4 μm) and correspond to modest misorientations, typically lower
324 than 5° (Figure S3). Detailed analysis reveals that the majority of subgrain boundaries are tilt walls,
325 i.e., with the rotation axis oriented nearly parallel to the subgrain wall. The most common subgrain
326 types for those three samples (deformed at 1050-1200 °C) are illustrated in Figure 8, where the
327 distribution of subgrain rotation axis is plotted in inverse pole figures. These plots reveal clustering
328 mostly around the [001] and $[\bar{1}10]$ directions. Subgrain rotation around $[\bar{1}10]$ is more prominent
329 at 1050 °C than at 1110 and 1200 °C. At 1200 °C, subgrain rotations around the $[\bar{1}10]$ direction
330 are negligible and rotations around [001] dominate, associated with a slightly larger contribution
331 from subgrain rotations around the <0kl> directions and around the [010] axis.

332 At 900-1000 °C fewer grains were analyzed. It is therefore difficult to identify specific rotation
333 axes as clearly as in the high-temperature samples (Figure S5). However, a similar trend is observed,
334 particularly for larger misorientation values (which present a lower uncertainty), with rotation axes
335 located near the $[\bar{1}10]$ and $[011]$ directions. We note that, unlike at 1050-1200 °C, those latter
336 rotations seem to be favored over those around the $[001]$ axis.

337 At 1050-1110 °C, the high concentration of subgrain rotations around $[001]$ probably reflects
338 the appearance of tilt walls as a result of dislocations of screw and edge character in the $[001](010)$
339 and $[100](010)$ slip systems, respectively, consistently with the CPOs described above. However,
340 other rotations, such as those around $[\bar{1}10]$ and $[011]$ observed throughout the entire 900-
341 1200 °C temperature range investigated (Figures S3 and S4), may simply reflect the presence of
342 GNDs. Those rotations can result from $[001]\{110\}$ and $[100]\{011\}$ dislocations, as already
343 identified in previous studies (Cordier et al., 2014; Demouchy et al., 2014; Thieme et al., 2018;
344 Wallis et al., 2016). Conclusively, misorientation may be used as a proxy for dislocation density in
345 general, but does not necessarily represent the density of mobile (free) dislocations accommodating
346 most of the strain.

347 The three samples investigated by TEM exhibit very distinctive microstructures, as shown
348 in Figure 9. At 950 °C (NF_950-1), consistently with the stress drop observed at high stress, one
349 observes mostly indication of a brittle response (Figure 9a). Although some intragranular
350 microcracks can be occasionally observed, brittleness results predominantly from grain boundary
351 cracking as seen in Figure 9b. In most cases, the grain boundary microcracks are straight and
352 cleavage-like. However, some exhibit serration, suggesting a different behavior before rupture
353 (Figure 9b, Figure S6a), characteristic of ductile cracks (Ponson et al., 2013). The intragranular
354 microstructure is very heterogeneous. Many grains are pristine, but dislocation activity can be
355 observed in some grains, probably related to microplasticity (i.e., plastic deformation by dislocation
356 motion occurs locally, while the bulk material remains essentially in the elastic/brittle field).

357 At the highest temperature investigated (NF_1200-1), the microstructure is also very
358 heterogeneous (Figure 9). Several grains are still dislocation free, however clear dislocation activity
359 is observed in many of them and, consistently with SEM observations, the grain boundaries are
360 perfectly cohesive (Figure 9h, Figure S6d). The most striking observations come from the sample
361 deformed at 1050 °C (NF_1050-1). Very marked grain boundary openings are ubiquitous
362 (Figures 9c-f). Contrary to NF_950-1, damage at grain boundary does not result from purely brittle
363 cleavage-like micro-fracturing. The shape of surfaces formed by crack opening clearly suggests
364 some ductile processes (Figure 9e, Figures S6b and S6c). As a result, some cohesion could be
365 maintained during crack opening. This is probably what explains the ductility exhibited by this
366 sample despite very strong damage. Another characteristic of this sample is the presence of
367 dislocation activity in almost all grains (Figure 9f), which is consistent with the GOS evolution
368 observed among samples (Figure 3). Although dislocation activity might be related to the ductile
369 processes suggested by the crack morphologies, no clear correlation could be evidenced.

370

371 **4. Discussion**

372 **Brittle to ductile transition mechanisms**

373 Between 900 and 1000 °C, no steady state could be achieved (Figure 2). Stress almost
374 immediately reached a yield point, beyond which deformation occurred in a semi-brittle manner,
375 via micro-cataclastic processes (intergranular cracks and sliding). As shown by the images of
376 recovered samples (Figures 4 and 9), for this range of temperature, the stress drop observed beyond
377 the stress maximum likely corresponds to the coalescence and propagation of pervasive micro-
378 crack networks. In the brittle field and across the brittle-ductile transition here, when grains slide
379 relative to each other (GBS), strain incompatibilities develop (usually at triple junctions), which can
380 lead to the formation of voids. In some cases, these voids further develop under local tensile
381 stresses and fully open grain boundaries (Figure 4, Figure 9d). Although these openings can be seen

382 as mode-I cracks in the brittle sample at 900 °C, an involvement of ductile processes in their
383 development is clearly identified at 950-1110 °C (Figures 4 and 9d,e), where they therefore
384 correspond to “ductile cracks” (Idrissi et al., 2016; Ponson et al., 2013). The presence of pervasive
385 gaping grain boundaries indicates indeed that deformation involved a significant contribution of
386 GBS at the sample scale (Ree, 1994, Langdon, 2006). This is well illustrated by the relative motion
387 of grains in Figure 4d and Figure 9c. The damage (i.e., cavitation) caused by this mechanism is a
388 direct consequence of the moderate confining pressure conditions (0.3 GPa) relative to stress levels
389 and the low temperature ($\sim 0.54\text{-}0.59 \times T_m$ for Fo_{100}), which, for our experimental durations,
390 prevents diffusion from filling the areas in tension as the grains are pulled apart (e.g., Ree, 1994).
391 Indeed, creep by diffusion would occur if characteristic diffusion lengths were greater than the
392 grain size of the aggregate. This criterion can be assessed using Si diffusivity since it is the slowest
393 diffusing species in silicates. For 900-1000 °C, data from Fei et al. (2012) predict lattice diffusivities
394 of 1.4×10^{-25} to $3.7 \times 10^{-24} \text{ m}^2 \text{ s}^{-1}$ and corresponding characteristic diffusion lengths of ~ 0.07 to
395 0.3 nm for the durations of our experiments, which excludes a significant contribution from
396 diffusion creep (e.g., Nabarro-Herring creep). Additionally, theoretical strain rates for diffusion
397 creep can be calculated from the diffusion coefficients mentioned above and using the maximum
398 (or steady-state) stress measured for each experiment (Raj and Ashby, 1971; Tasaka et al., 2013).
399 Using a grain size of 3 μm , representative of our samples, these calculations yield strain rates of the
400 order of 10^{-13} - 10^{-10} s^{-1} and therefore confirm the unlikely dominance of diffusion creep in our
401 experiments.

402 Interestingly, these low-temperature samples ($< 1000 \text{ }^\circ\text{C}$) sustained major strain hardening
403 without reaching failure and largely exceeded the Goetze criterion, i.e., differential stress exceeded
404 confining pressure (Kohlstedt et al., 1995). Transient violation of the Goetze criterion has been
405 previously reported in similar deformation experiments on fined-grained iron-bearing olivine
406 (Demouchy et al., 2009, 2014; Thieme et al., 2018). In the present case, the Goetze criterion should

407 only be seen as a condition that predicts the nucleation of cracks (i.e., cavitation), but without
408 necessarily resulting in macroscopic failure.

409 Interestingly, SEM images of our low-temperature samples show more pronounced surface
410 relief than samples deformed at $> 1100\text{ }^{\circ}\text{C}$ (Figure 4b). These features are very similar to the out-
411 of-plane grain displacements on free surfaces reported by Bollinger et al. (2019) at 3.5-5 GPa (1000-
412 $1200\text{ }^{\circ}\text{C}$, $2 \times 10^{-5}\text{ s}^{-1}$), as well as to those evidenced on samples deformed by GBS creep at 1 atm
413 (Watanabe et al., 1982; Maruyama and Hiraga, 2017a, 2017b; Masuda et al., 2015, 2016). Although,
414 the physical explanation for the appearance of these displacements on free-surfaces created after
415 the experiments remains elusive. It is suspected that this late relative grain motion occurs as a
416 relaxation process at grain boundaries in response to large residual stress heterogeneities due to
417 stored elastic energy. This relaxation is made possible by the decohesion of a significant fraction
418 of the grain boundaries. The present study and those of Maruyama and Hiraga (2017a) and
419 Bollinger et al. (2019) thus show the possible activation of GBS under a wide range of confining
420 pressure (0.3-5 GPa). We argue that, under relatively low temperature, rapid laboratory strain rates
421 generate heterogeneous stress concentrations, which can locally be at least equal to the confining
422 pressure, even under several gigapascals of pressure (Druiventak et al., 2011; Burnley, 2013). These
423 stress concentrations either create openings (cavitation), as in our case, or, at high temperature,
424 drive ionic diffusive fluxes towards low-pressure areas (e.g., Wheeler, 2018).

425 **Grain boundary mediated deformation at low strain**

426 The apparent Young moduli (Figure 2b, Figure S2) provide valuable information about the
427 deformation behavior of the aggregates at low strain. Both yield stresses and corresponding
428 apparent Young moduli are reported in Figure 10 as a function of temperature. Our results describe
429 a linear temperature dependence of $-263\text{ MPa}/^{\circ}\text{C}$. This value is too large to be explained by
430 forsterite elasticity only, as shown by the comparison with the values of Suzuki et al. (1983), which
431 yield a temperature derivative of $-32\text{ MPa}/^{\circ}\text{C}$ (Figure 10a). These results corroborate the recent

432 study of Burnley and Kaboli (2019) on iron-bearing olivine aggregates. It is unclear what percentage
433 of the strain is accommodated elastically or plastically at this stage of the deformation curves. This
434 deviation from purely elastic behavior observed upon modest amounts of strain may reflect a
435 contribution of GBS, accordingly with TEM images, which evidence an involvement of ductile
436 processes in the grain boundary network. In any case, these data suggest a major rheological impact
437 of the grain boundaries with increasing temperature. Since grain boundary mediated deformation
438 is activated at lower temperatures than crystal plastic mechanisms, it may play a key role in the
439 mantle at low temperature and/or high stress conditions, typical of the brittle-ductile transition.

440

441 **Flow mechanisms and comparison to previous rheological laws**

442 In the 900-1000 °C range, there is no deformation-induced CPO pattern (Figure 7),
443 accordingly with the low amounts of strain experienced by the samples. At the TEM scale,
444 dislocation activity is observed in NF_950-1, but is likely related to microplasticity, and coexists
445 with pervasive microfracturing (Figure 9a). As discussed above, in this temperature range,
446 deformation is transiently ductile at the sample scale, and shortening results from the propagation
447 of micro-cracks at the grain (micrometric) scale, together with minor dislocation activity (glide of
448 [100] and [001] dislocations), as previously proposed by Druiventak et al. (2011). Consequently, the
449 maximum stresses obtained in this temperature range correspond to a semi-brittle regime and lie
450 largely above previously established low temperature flow laws, as shown in Figure 11. We note,
451 as pointed out by previous studies (e.g., Demouchy et al., 2009, 2013, 2014, Thieme et al., 2018),
452 that extrapolations from power law flow yield overestimated stress values for the present
453 temperature range and strain rates.

454 At, or above, 1050 °C, apparent steady state was achieved over the strain range investigated
455 (Figure 2) and two observations stand out: (1) GOS values are the highest at 1100 °C, then decrease
456 at 1200 °C (Figure 3). Therefore, the increase in GOS from 900 to 1100 °C, associated with the

457 appearance of numerous subgrain boundaries (Figure 8), reflects an increasing contribution of
458 intracrystalline plasticity with temperature, whereas the lower value obtained at 1200 °C likely
459 indicates a contribution of diffusive mechanisms. (2) In addition, grain boundary sliding, opening
460 and serration are also commonly observed (Figure 4, and Figures 9c-e), indicative of grain boundary
461 mediated plasticity. Thus, deformation in the 1050-1110 °C range corresponds to a transitional
462 regime where both plastic and cataclastic deformation mechanisms are active.

463 At 1200 °C, a quasi-steady state is achieved rapidly, after only 3 % of strain. The
464 microstructure is noticeably different, with almost no grain pluck out, no evidence of grains sliding
465 relative to each other, no out-of-plane motion, and less grain boundary serration, when compared
466 to samples deformed at 1050 and 1110 °C. Grain boundaries are preserved and appear more curved
467 than at lower temperature; and boundary bulging is clearly identified (Figure 4h). Together with a
468 small decrease in GOS and weaker olivine CPO patterns, these observations suggest further
469 activation of recovery mechanisms (subgrain boundary formation, grain boundary migration, ionic
470 diffusion) than at lower temperature. This is consistent with recent values of Si diffusion in
471 forsterite (Fei and Katsura, 2016), which, at 1200 °C, predict a strain rate of $\sim 10^{-5} \text{ s}^{-1}$ for a grain
472 size of 1 μm and a stress of $\sim 500 \text{ MPa}$ (Raj and Ashby 1971). Therefore, as already observed by
473 Tasaka et al. (2013) under the same pressure, temperature and strain rate conditions, our
474 experiment at 1200 °C also points to a transitional regime where both dislocation-accommodated
475 grain boundary migration and diffusive mechanisms are active.

476 Conclusively, our results report a mechanical continuum from low temperature (i.e., semi-
477 brittle) to high temperature plasticity. The temperature dependence of stress obtained at $\geq 1050 \text{ °C}$
478 (Figure 11) is in agreement with that calculated from power law flow (e.g., Hirth and Kohlstedt,
479 2003; Ohuchi et al., 2015). Below 1050 °C, however, the temperature-dependence is greater than
480 that calculated from exponential laws either from experimental studies (e.g., Evans and Goetze,
481 1979; Demouchy et al., 2013; Faul et al., 2011) or numerical modeling (Boioli et al., 2015b). As

482 detailed in the above, this is due to the occurrence of a variety of deformation mechanisms at play
483 throughout the temperature range investigated.

484

485 **Effect of iron on olivine strength**

486 The stress values obtained in the present study are slightly larger (~ 17 and 50% at 1000
487 and $1200\text{ }^\circ\text{C}$, respectively) than those reported for San Carlos olivine (Fo_{90}) aggregates in Thieme
488 et al., (2018) using the same apparatus, under identical PT and strain rate conditions and with a
489 comparable grain size (Figure 11), which is qualitatively consistent with the notorious weakening
490 effect of iron on olivine (Bollinger et al., 2015; Zhao et al., 2009). The compositional effect
491 observed here is weaker than the one reported by Zhao et al., (2009) at identical PT conditions,
492 which predicts at least an order of magnitude increase in stress. However, their iron-content
493 dependent rheology law was based on Fo_{00} - Fo_{90} compositions and the chosen expression is
494 mathematically unfit to predict stress/strain values for Fo_{100} (strain rate nil for Fo_{100}). A direct
495 rheological comparison between iron-bearing and iron-free olivine at high pressure (3.1 - 8.1 GPa)
496 by Bollinger et al. (2015) has also reported weaker effects of iron incorporation. Following the
497 approach of Bollinger et al., (2015, see their equation 3), i.e., using a power flow law and assuming
498 constant activation energies and pre-exponential terms; the difference observed at $1200\text{ }^\circ\text{C}$ can be
499 explained by a stress exponent decrease from 3.5 for Fo_{90} olivine to only ~ 3.25 for Fo_{100} forsterite,
500 whereas an exponent of 2.3 was found by Bollinger et al. (2015) at 1100 - $1300\text{ }^\circ\text{C}$ and $\sim 5\text{ GPa}$. The
501 present results thus point to a small rheological effect of iron (between Fo_{100} and Fo_{90}) on GBS in
502 very fine-grained olivine aggregates.

503

504 **Implications for the lithospheric mantle**

505 In summary, the GBS observed in our samples in the brittle-ductile transition field (950-
506 1050 °C) upon modest amounts of strain (<2 %), is first transiently accommodated elastically and
507 then viscously. Upon further strain, GBS eventually produces significant cavitation. This reflects
508 specific experimental conditions, namely (i) relatively low temperatures ($T/T_m \leq 0.68$), (ii) fast
509 strain rates (10^{-5} s^{-1}) and (iii) sub-micron grain-sized and well annealed polygonal microstructures,
510 which are all prerequisites to generate high macroscopic differential stress relative to confining
511 pressure and induce cavitation without immediate failure of the samples. Such conditions may be
512 achieved in mantle shear zones with peculiar settings, such as in tectonically exhumed mantle,
513 where deformation at crustal pressure and temperatures may locally allow stress to exceed
514 lithostatic pressure and, in turn, lead to GBS and gaping as observed in our experimental study.
515 Under the present dry conditions, high local differential stresses are needed for cavitation to
516 generate unpressurized voids (Burnley, 2013). However, cavitation may occur with lower stress
517 levels if pressurized aqueous fluids or reactive melts are present (Précigout and Stünitz, 2016).

518 In addition, the predominance of diffusion creep or GBS over dislocation creep in
519 mylonitic shear bands in peridotites is still debated based on field and microstructural evidences. It
520 strongly depends on stress and strain rate evolution upon grain size reduction (Platt and Behr,
521 2011). On the other hand, the recent review of Vauchez et al. (2012) suggests that, in most
522 instances, the dominant mechanism remains dislocation creep, associated to grains sizes of typically
523 $\sim 200 \mu\text{m}$. In any case, in the lithospheric mantle, GBS and cavitation might be transient
524 mechanisms occurring in incipient mantle shear zones in response to large stresses. Eventually,
525 lower strain rates and stresses will likely allow ionic diffusion, rather than cavitation, to
526 accommodate deformation.

527

528 5. Conclusions

- 529 (1) In the present experiments, several deformation mechanisms are at play with their
530 respective contributions varying with temperature, and stress levels. We document a
531 transition from micro-cracking to grain boundary sliding, and then to dislocation
532 creep at around 1050-1100 °C; with a potential important contribution of ionic
533 diffusion at 1200 °C.
- 534 (2) The transition results in subtle changes in SPO and CPO, but is evident in the
535 recovered microstructures in SEM and TEM. Moreover, in our experiments, as well
536 as in previous experimental studies, grains slide against their neighbors towards areas
537 where, local tensile stresses have been generated by large stress heterogeneities. This
538 is possible under moderate confining pressure in laboratory, where the Goetze
539 criterion can be violated in a transient way.
- 540 (3) Given the experimental high-stress conditions, our results may not directly apply to
541 the deformation of the Earth's mantle at large scale. Nevertheless, our results
542 highlight that the grain boundary network in forsterite aggregates is an agent of
543 deformation that is activated at lithospheric temperatures (< 1100 °C), before
544 intracrystalline deformation.

545

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559

560 7. References

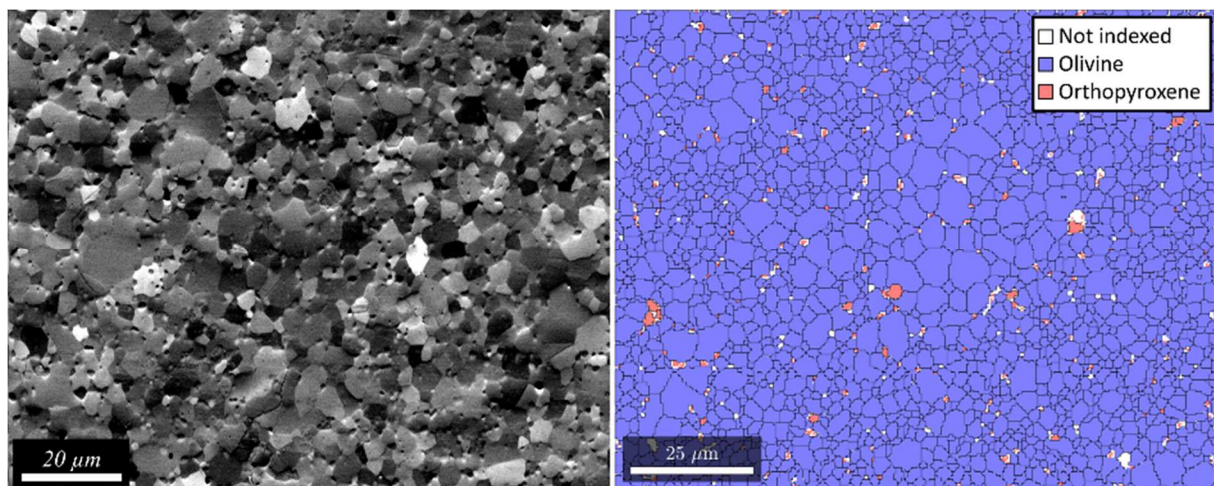
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- 718

719 **Figures**

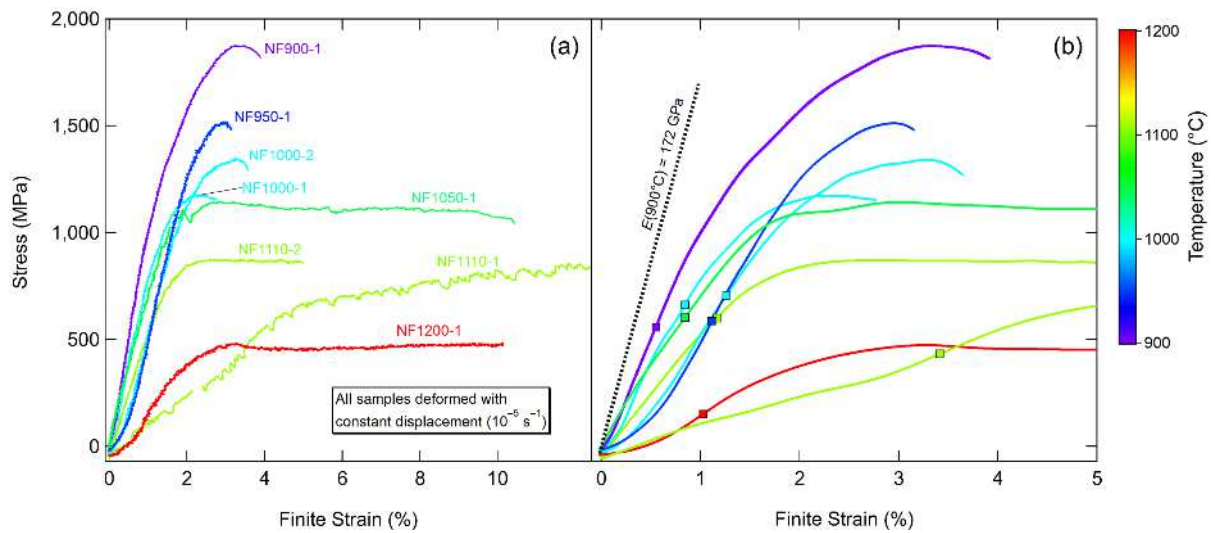


720

721 Figure 1: (a) SEM image (Forward scattered electrons) of the starting material used for the deformation
722 experiments. (b) EBSD map collected on the same starting material showing phase distribution and grain
723 boundaries determined with MTEX (black lines, see text for details).

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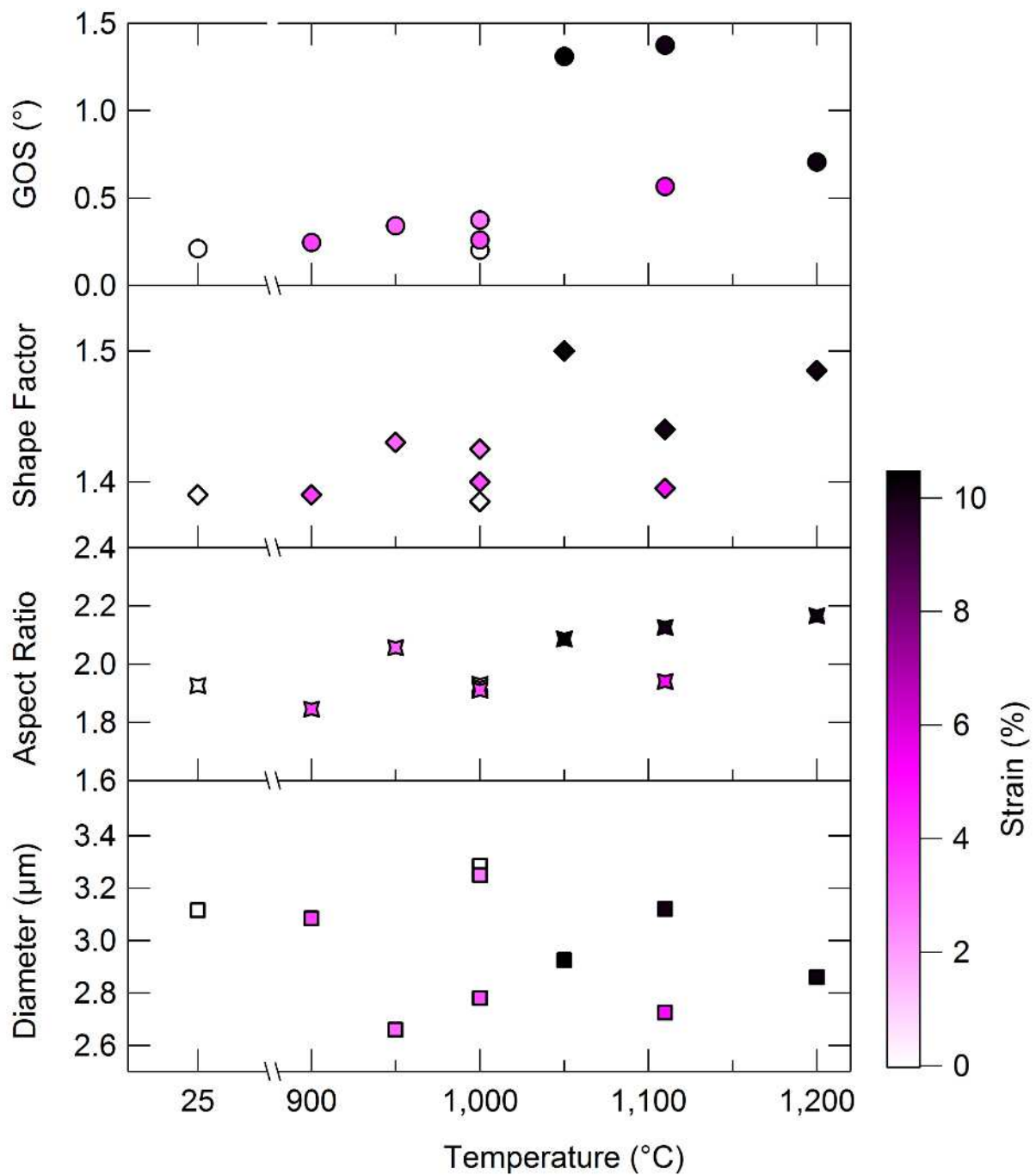
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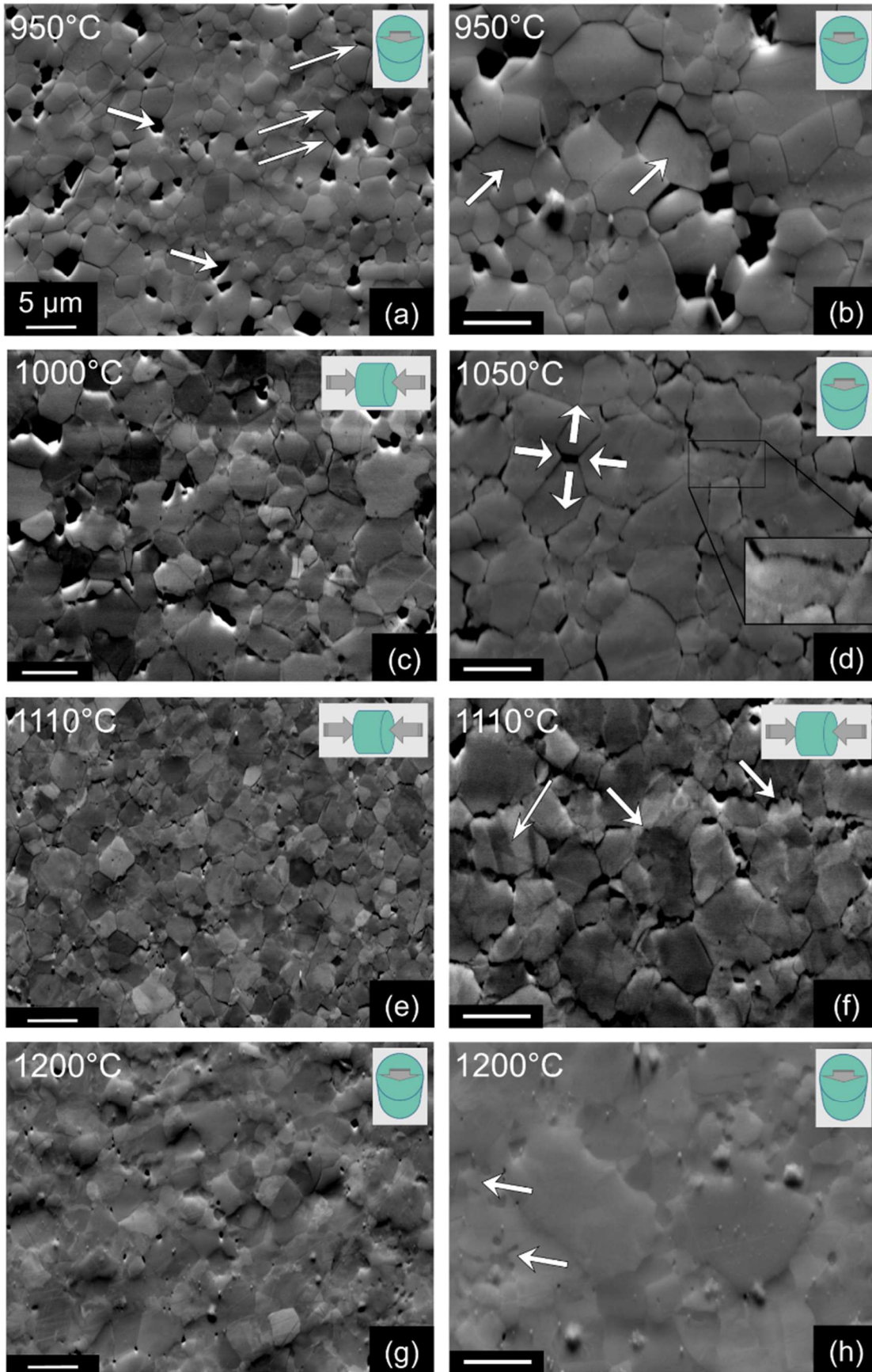
727 Figure 2: Stress-strain curves for axial deformation experiments. Each curve is colored as a function of
728 temperature, according to the appended color scale. (a) Mechanical curves after data treatment (see section
729 2.2 for details). (b) Smoothed stress-strain curves (up to 5% strain) used to retrieve yield points, indicated
730 by squares (see supp. mat. for details). As a guide to the eye, the Young modulus of forsterite at 900°C
731 (172 GPa), calculated using the data of Suzuki et al. (1983), is also shown next to the data of NF_900-1, for
732 which the apparent Young modulus is 112.2 GPa.

733



735

736 Figure 3: Grain size, shape parameters (shape factor and aspect ratio) and grain orientation spread (GOS)
 737 as a function of temperature and finite strain. The leftmost datum point at room temperature and no strain
 738 corresponds to the starting material. The other nine data points correspond to the static annealing test
 739 (1000 °C, no strain) and the axial deformation experiments.

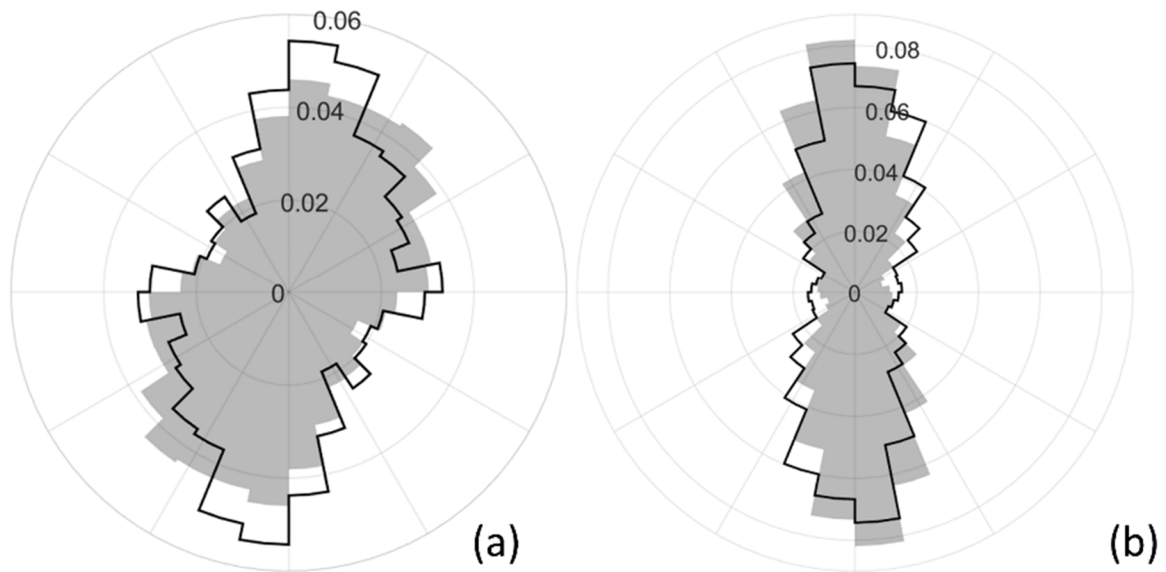


740

741 Figure 4: SEM images (forward scattered electrons) of samples deformed at various temperatures. Scale bar
 742 is 5 μm on all images. The direction of the deformation (either horizontal or in-plane) is given by the

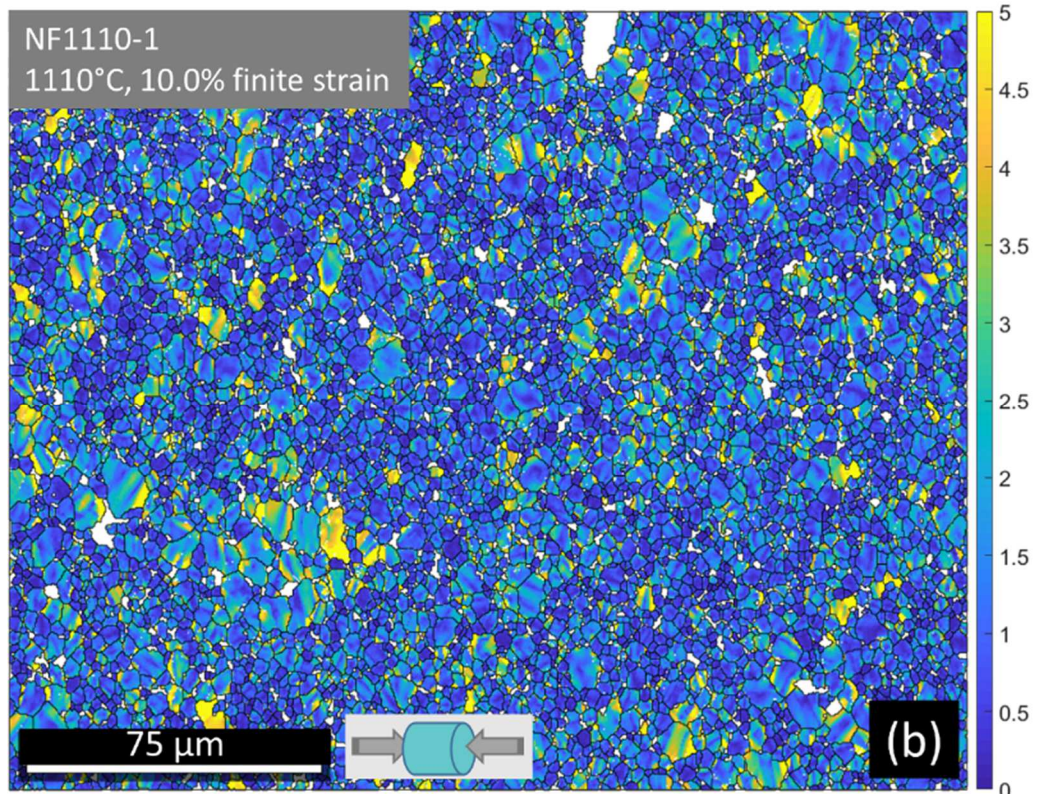
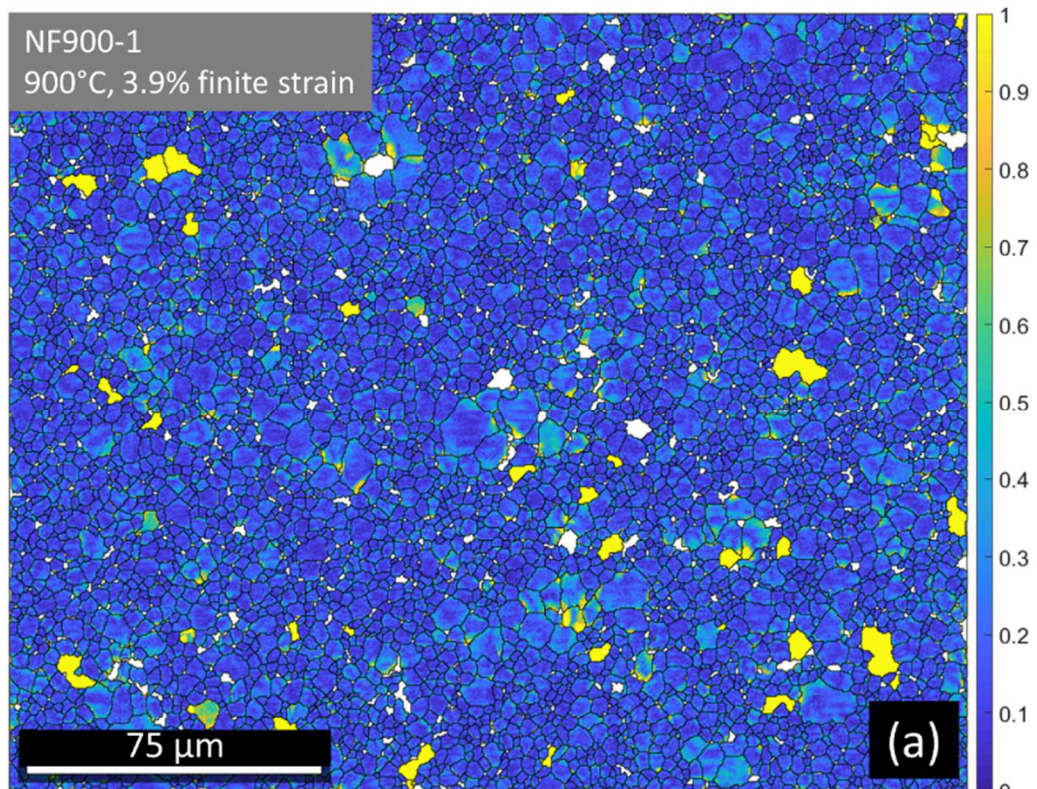
743 schematic drawing in the top right corner of each image. (a) NF_950-1, 3.1% finite strain. Gaping grain
744 boundaries (thin white arrows) and grain plucking due to cross-section preparation (thick white arrows) are
745 visible. (b) NF_950-1. Arrows point at two grains, which show a significant vertical offset, as evidenced by
746 the shadows on the top of the grains. (c) NF_1000-2, finite strain of 3.6%. (d) NF_1050-1, finite strain of
747 10.4 %. The inset is a higher-magnification image with enhanced contrast that better shows serrated grain
748 boundaries. The white arrows depict relative grain motion, which has left a void between pulled-apart grains.
749 (e) NF_1110-2, finite strain of 5 %. (f) NF_1110-1, finite strain of 12.41 %. Thick arrows show serrated
750 grain boundaries, which have been characterized in detail with TEM (see below for more details). The
751 thinner arrow indicates a grain where bands with variable contrast highlight subgrain boundaries and thus
752 intracrystalline plasticity. (g) NF_1200-1, finite strain of 10 %. (h) NF_1200-1. Arrows show migrating (i.e.,
753 bulging) grain boundaries.

754



756

757 Figure 5: Polar histograms displaying the normalized orientation distribution of the longest axis of forsterite
 758 grains. Samples length, and therefore axial compression direction, is horizontal. (a) Starting material (black
 759 line) and sample NF_900-1 (grey bars), deformed to a finite strain of 3.9 %. (b) Samples NF_1110-2 (black
 760 line) and NF_1110-1 (grey bars) deformed by 5 and 12.41 % of finite strain, respectively.



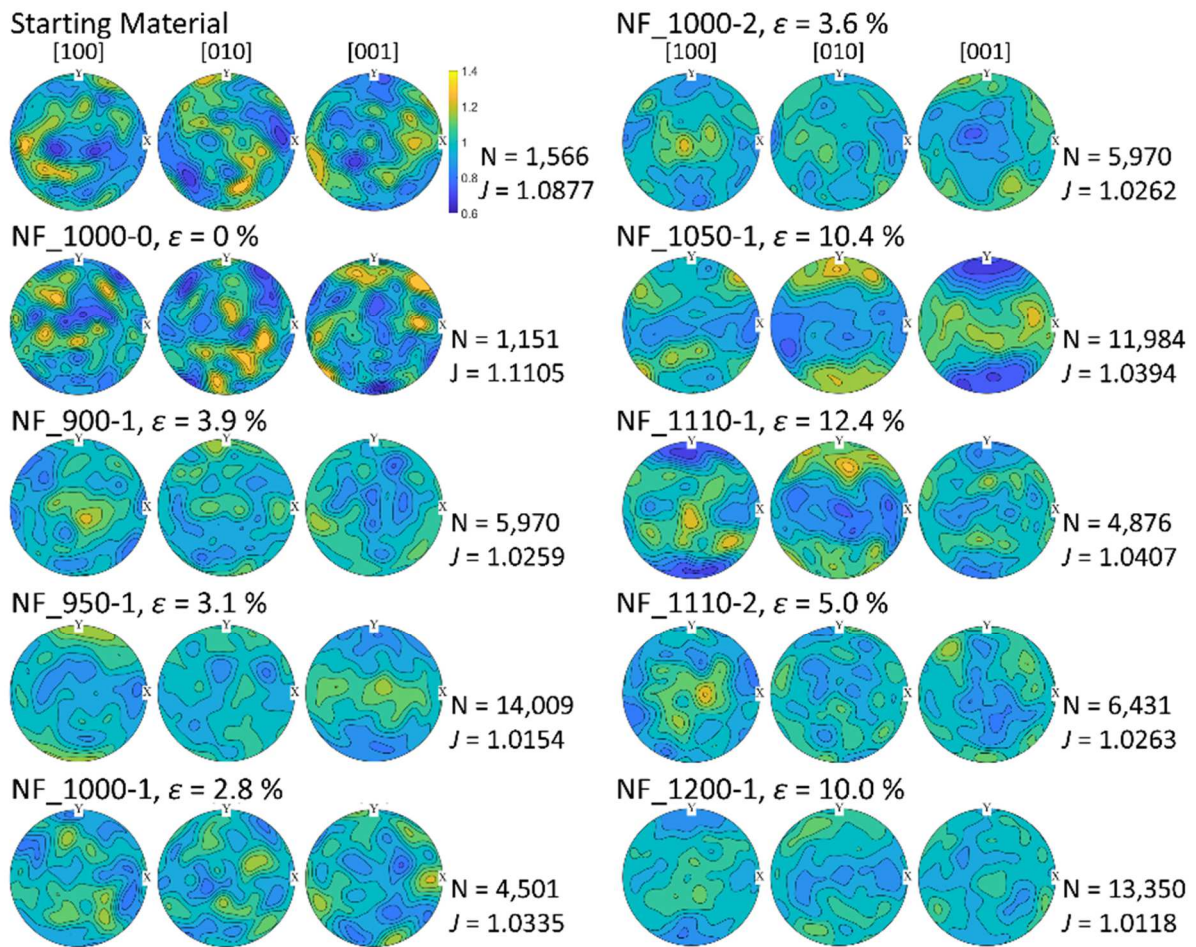
761

762 Figure 6: EBSD maps showing grain boundaries and Mis2Mean angles ($^{\circ}$) within each grain. (a) Sample

763 NF_900-1 (3.9 % finite strain). Very limited grain plasticity is observed, as shown by Mis2Mean values,

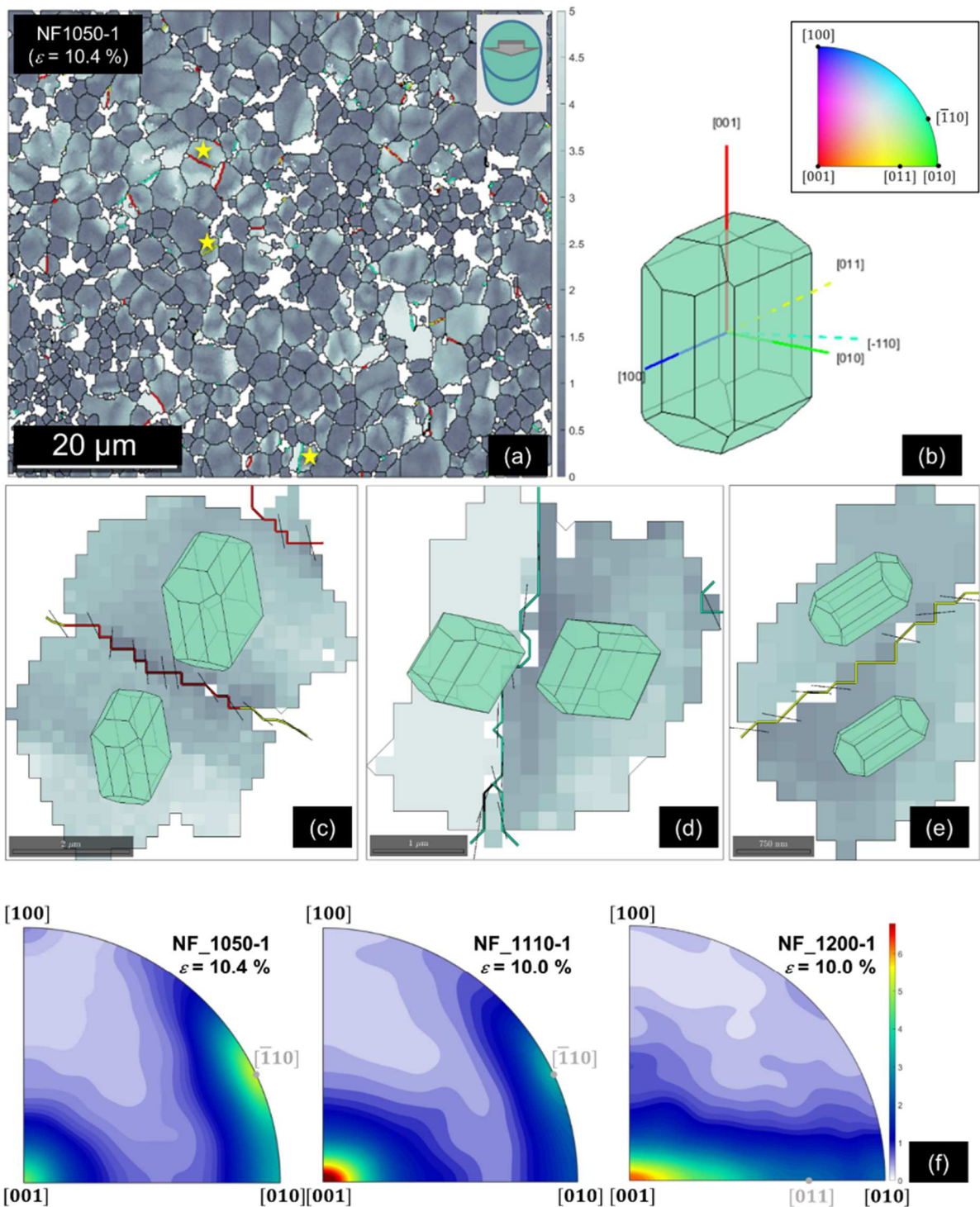
764 which rarely exceed 1° . (b) Sample NF_1110-1 (12.41 % finite strain), which presents the largest GOS values

765 observed in our samples. Note that the color scale maximum in (b) is five times larger than in (a).



766

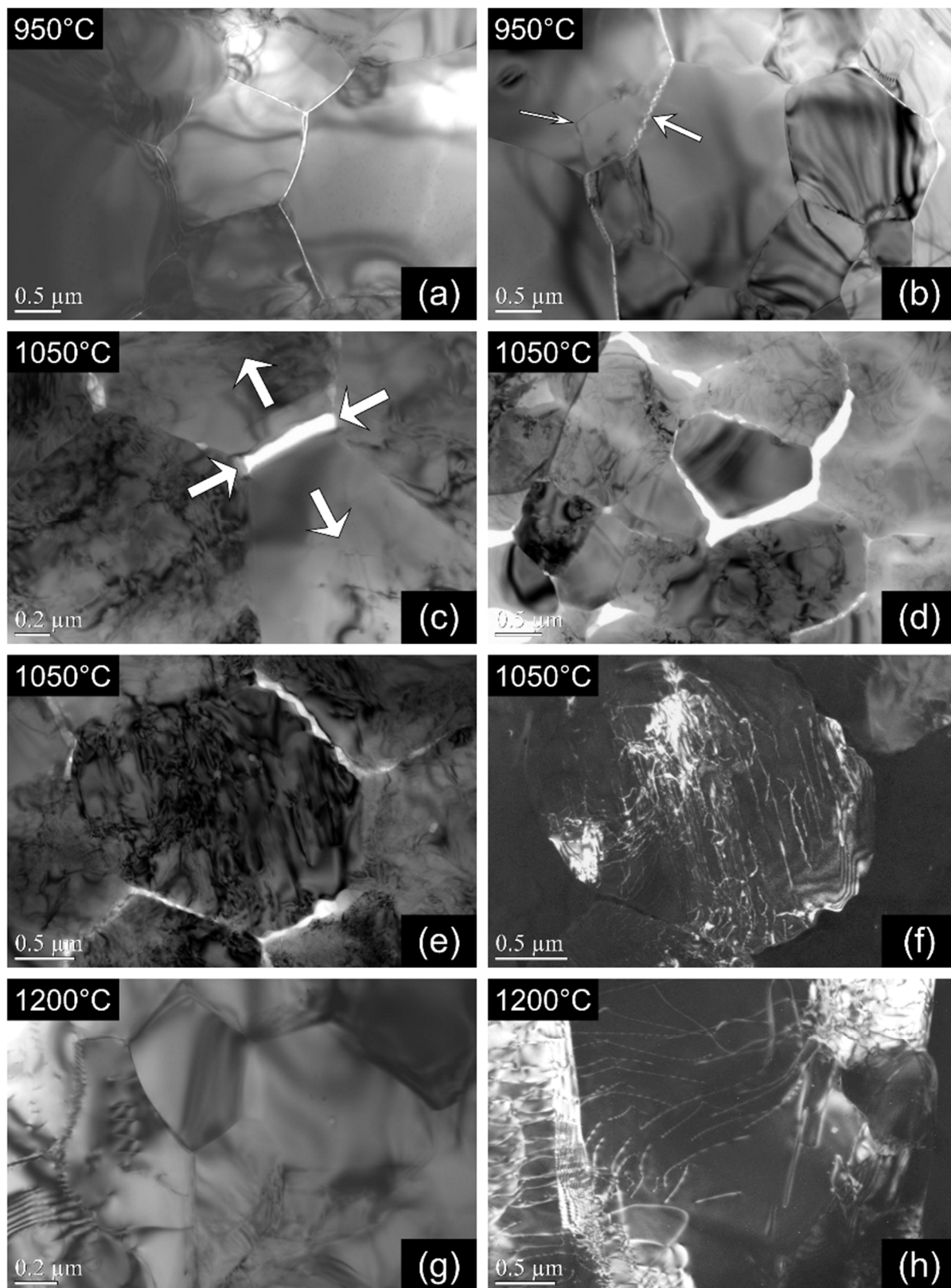
767 Figure 7: Pole figures of forsterite obtained for the starting material, the annealing experiment and each axial
 768 deformation experiment. Densities were obtained from the largest EBSD map collected on each sample
 769 and reduced to one point per grain. Densities were contoured with a 0.06 interval. The color scale given
 770 beside the starting material pole figures is the same on all figures. The number of grains used, N, and the
 771 corresponding J-index are provided in each case. The direction of axial compression deformation is vertical
 772 for all figures, except for sample NF_900-1, where orientation was lost due brittle failure of the sample.



773

774 Figure 8: Intracrystalline plasticity at 1050-1200 °C. (a)-(e) Misorientation analysis in sample NF_1050-1. (a)
 775 EBSD map showing Mis2Mean values, grain boundaries (black lines) and subgrain boundaries (thick colored
 776 lines). (b) Reference forsterite crystal habitus used in (c), (d) and (e). In addition to principal axes, specific
 777 axes, corresponding to $\bar{1}10$ and 011 subgrain rotations, are shown and colored according to the inverse
 778 pole figure colors given in the inset. (c)-(e) Detailed views of grains (and subgrains) marked by a star in (a).
 779 The bars on top of the subgrain boundaries represent the orientation of the subgrain rotation axis; their
 780 length is a function of their dipping angle (i.e., they are shorter when the rotation axis sticks out of plane).

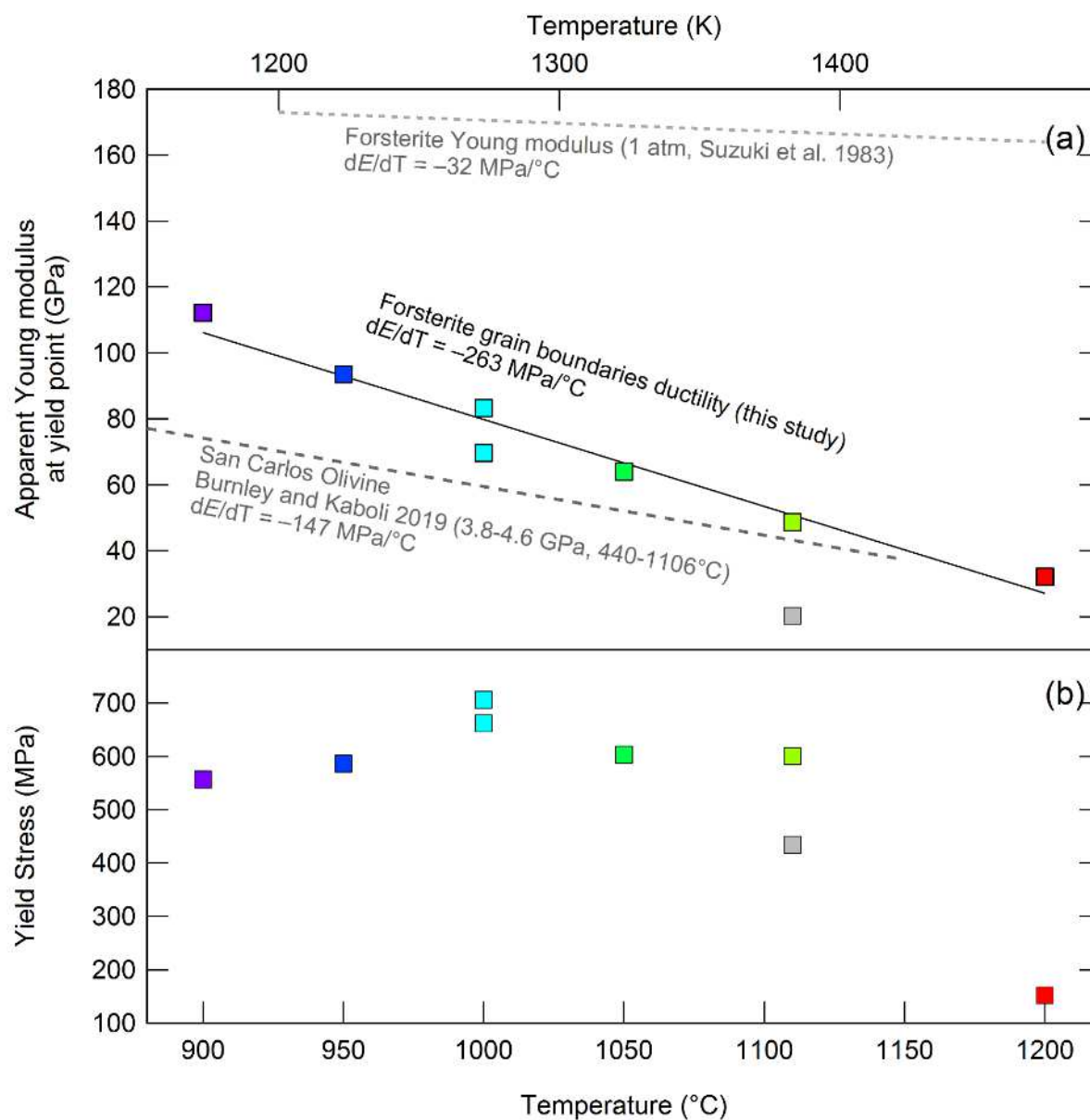
781 Similar figures for samples NF_1110-1 and NF_1200-1 are provided in the supplementary material (Figures
782 S3 and S4). (f) Inverse pole figures displaying the densities of subgrains rotation axes for samples NF_1050-
783 1, NF_1110-1 and NF_1200-1. The color scale gives the axis density. The majority of subgrain rotations
784 occur around the [001] and $[\bar{1}10]$ axis, and occasionally around the [011] axis; this latter case being more
785 common in sample NF_1200-1.



786

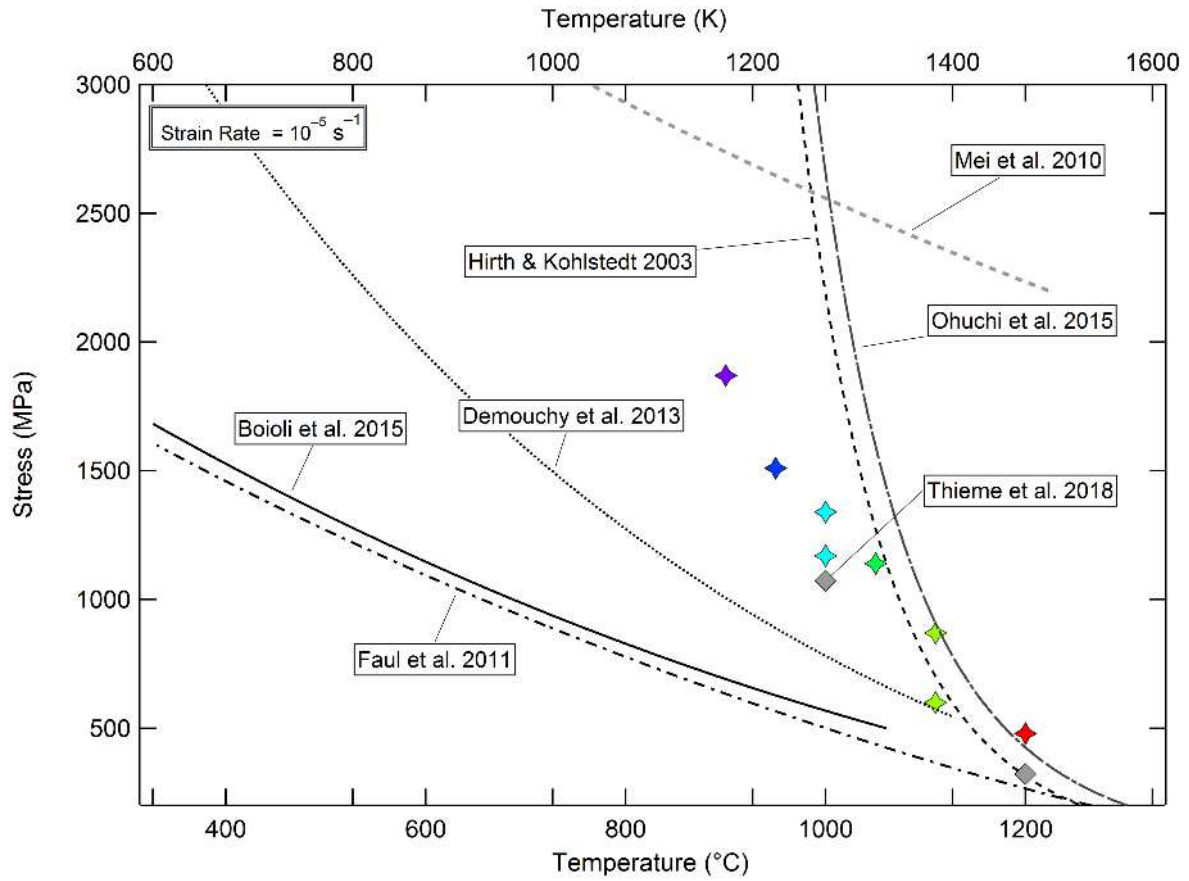
787 Figure 9: TEM images of samples deformed at 950-1200 °C. (a)-(b) Sample NF_950-1. (a) Grains show few
 788 to no dislocation. Gaping grain boundaries are straight, typical of brittle failure. (b) Several grain boundaries
 789 are serrated at the scale of a few tens of nanometers (thick arrow) and intragranular cracks are also present
 790 (thin arrow). A higher magnification image of these features is provided in the supplementary material

791 (Figure S6a). (c)-(f) Sample NF_1050-1. (c) Similar to SEM observations, cavitation and relative grain
792 motion (white arrows) is observed. (d) In most cases the voids created upon deformation leave a mismatch
793 between the grains pulled apart, i.e., the void cannot be closed by simply bringing the grains back together,
794 thus suggesting a role of intracrystalline plasticity. (e) Opened grain boundaries are serrated at a larger scale.
795 (f) Dark field imaging of the same grain reveals a high dislocation density. (g)-(h) Sample NF_1200-1. (g)
796 Grain boundaries remained closed and, in certain areas, grains are pristine and almost dislocation free. (h)
797 Dark field image in another area, where grains reveal high dislocation densities. Dislocation sources (loops)
798 are visible in the middle lower part of the image.



800

801 Figure 10: (a) Apparent Young modulus at yield point as a function of temperature. Young modulus of
 802 forsterite as a function of temperature from Suzuki et al. (1983) is shown for comparison. Values of grain
 803 boundaries apparent elasticity for iron-bearing olivine from Burnley and Kaboli (2019) are also reported. (b)
 804 Yield stress as a function of temperature from the mechanical curves displayed in Figure 2b (see Supp Mat
 805 for details, Figure S2).



806

807 Figure 11: Comparison of iron-bearing olivine strength from the literature with the mechanical data from
 808 this study obtained on forsterite. All data are for a strain rate of 10^{-5} s^{-1} . The data points were colored as a
 809 function of temperature, according to same color scale used in Figure 2.

		Starting Material		Static Annealing		Deformation Experiments															
	Sample Name	SM		NF1000-0		NF900-1		NF950-1		NF1000-1		NF1000-2		NF1050-1		NF1110-1		NF1110-2		NF1200-1	
Experimental Conditions and Results	Temperature (°C)	NA		1,000		900		950		1,000		1,000		1,050		1,110		1,110		1,200	
	Sleeve Type	NA		Ag		Ag		Ag		Cu		Cu		Cu		Ni		Ni		Ni	
	Initial Length (mm)	NA		NA		7.25		10.20		10.35		9.60		10.55		10.42		9.48		10.35	
	Strain (%)	NA		NA		3.9		3.1		2.8		3.6		10.4		12.4		5.0		10.0	
	Duration (s)	NA		14,400		9,308		6,502		5,154		8,198		12,668		14,024		6,917		11,022	
	Max Strength (MPa)	NA		NA		1,870		1,510		1,170		1,340		1,140		840		870		480	
	Yield Stress (MPa)	NA		NA		557		586		662		706		603		434		600		152	
	Apparent <i>E</i> at Yield (GPa)	NA		NA		112.2		93.5		83.2		69.6		63.9		20.2		48.6		32.1	
EBSD Analysis	# Grains	1,566	1,035	1,151	1,077	4,625	5,970	1,544	14,009	4,501	4,185	5,970	1,564	11,984	823	4,876	4,628	6,431	6,054	13,350	951
	<i>J</i> index	1.1637	1.2680	1.2526	1.2472	1.0618	1.0509	1.1838	1.0236	1.0782	1.0871	1.0481	1.1568	1.0532	1.3567	1.0657	1.0827	1.0449	1.0432	1.0235	1.3568

	J index 1PPG	1.0 87 7	1.1 28 5	1.1 10 5	1.1 33 5	1.0 31 4	1.0 25 9	1.0 94 9	1.0 15 4	1.0 33 5	1.0 36 4	1.02 62	1.0 85 7	1.0 39 4	1.2 43 4	1.0 40 7	1.0 45 3	1.0 26 3	1.0 24 6	1.01 18	1.1 56 9
	Average Grain Size (μm)	3.1 15		3.2 85		3.0 85		2.6 60		3.2 50		2.78 0		2.9 25		3.1 20		2.7 25		2.86 0	
	Mean Aspect Ratio	1.9 25		1.9 30		1.8 45		2.0 55		1.9 20		1.91 0		2.0 85		2.1 25		1.9 40		2.16 5	
	Mean Shape Factor	1.3 90		1.3 85		1.3 90		1.4 30		1.4 25		1.40 0		1.5 00		1.4 40		1.3 95		1.48 5	
	Mean GOS ($^{\circ}$)	0.2 10		0.2 00		0.2 45		0.3 40		0.3 75		0.26 0		1.3 10		1.3 75		0.5 65		0.70 5	