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

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Abstract

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

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

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

1. Introduction

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

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

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

3

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

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

2. Materials and Methods

2.1 Starting material

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

3. Results

3.1 Mechanical data

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

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

3.2 Microstructures

Results from EBSD analyses on the deformed aggregates are summarized in Table 1. Average 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 °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 °C. Visually,

226 textures from samples deformed more extensively and at greater temperatures (1050-1200° 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 °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 °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 °C and which is not observed at

233 1200 °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 ~45° 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_110-1. 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 ~45° with respect to the compression direction, respectively. These

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

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

16

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 (~0.54-0.59 × Tm 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 3.7×10^{-24} m^2 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 °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 °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 °C, 2×10^{-5} 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 stain 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 MPa/°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 MPa/°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

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

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

Conclusively, our results report a mechanical continuum from low temperature (i.e., semi-brittle) to high temperature plasticity. The temperature dependence of stress obtained at $\geq$ 1050 °C (Figure 11) is in agreement with that calculated from power law flow (e.g., Hirth and Kohlstedt, 2003; Ohuchi et al., 2015). Below 1050 °C, however, the temperature-dependence is greater than that calculated from exponential laws either from experimental studies (e.g., Evans and Goetze, 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 °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 ion-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 °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 °C and ~5 GPa. The

501 present results thus point to a small rheological effect of iron (between Fo_{100} and F_{90}) on GBS in

502 very fine-grained olivine aggregates.

503

504 **Implications for the lithospheric mantle**

21

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   ~200 µm. 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.

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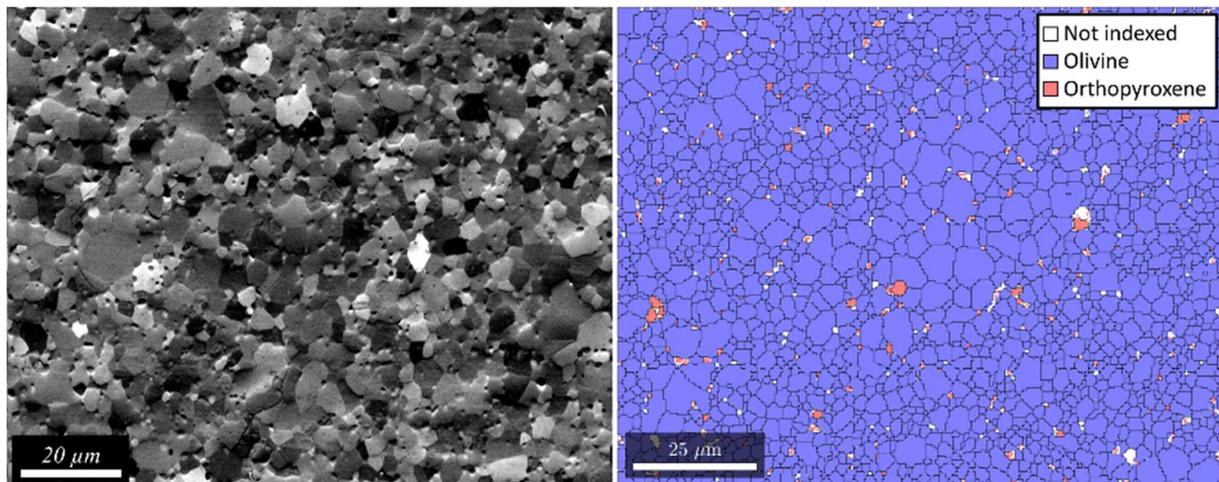
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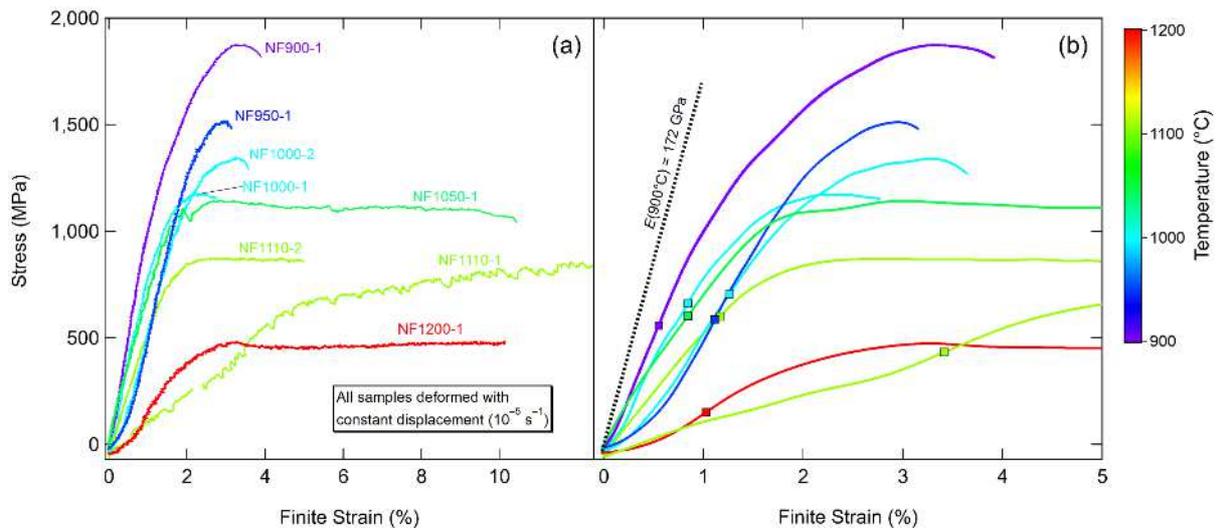
Figure 1: (a) SEM image (Forward scattered electrons) of the starting material used for the deformation experiments. (b) EBSD map collected on the same starting material showing phase distribution and grain boundaries determined with MTEX (black lines, see text for details).

725

726

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

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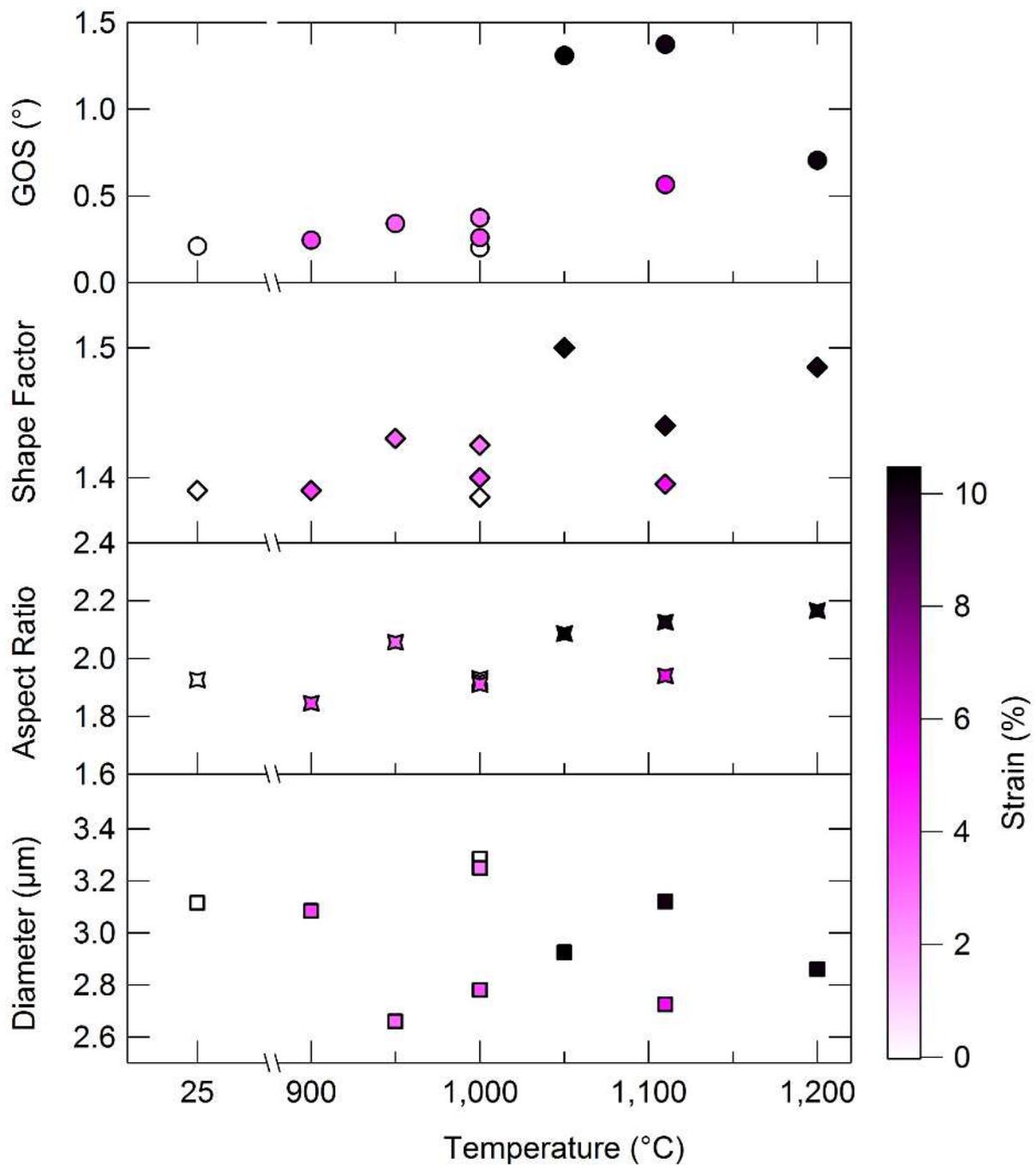
Figure 3: Grain size, shape parameters (shape factor and aspect ratio) and grain orientation spread (GOS) as a function of temperature and finite strain. The leftmost datum point at room temperature and no strain corresponds to the starting material. The other nine data points correspond to the static annealing test (1000 °C, no strain) and the axial deformation experiments.
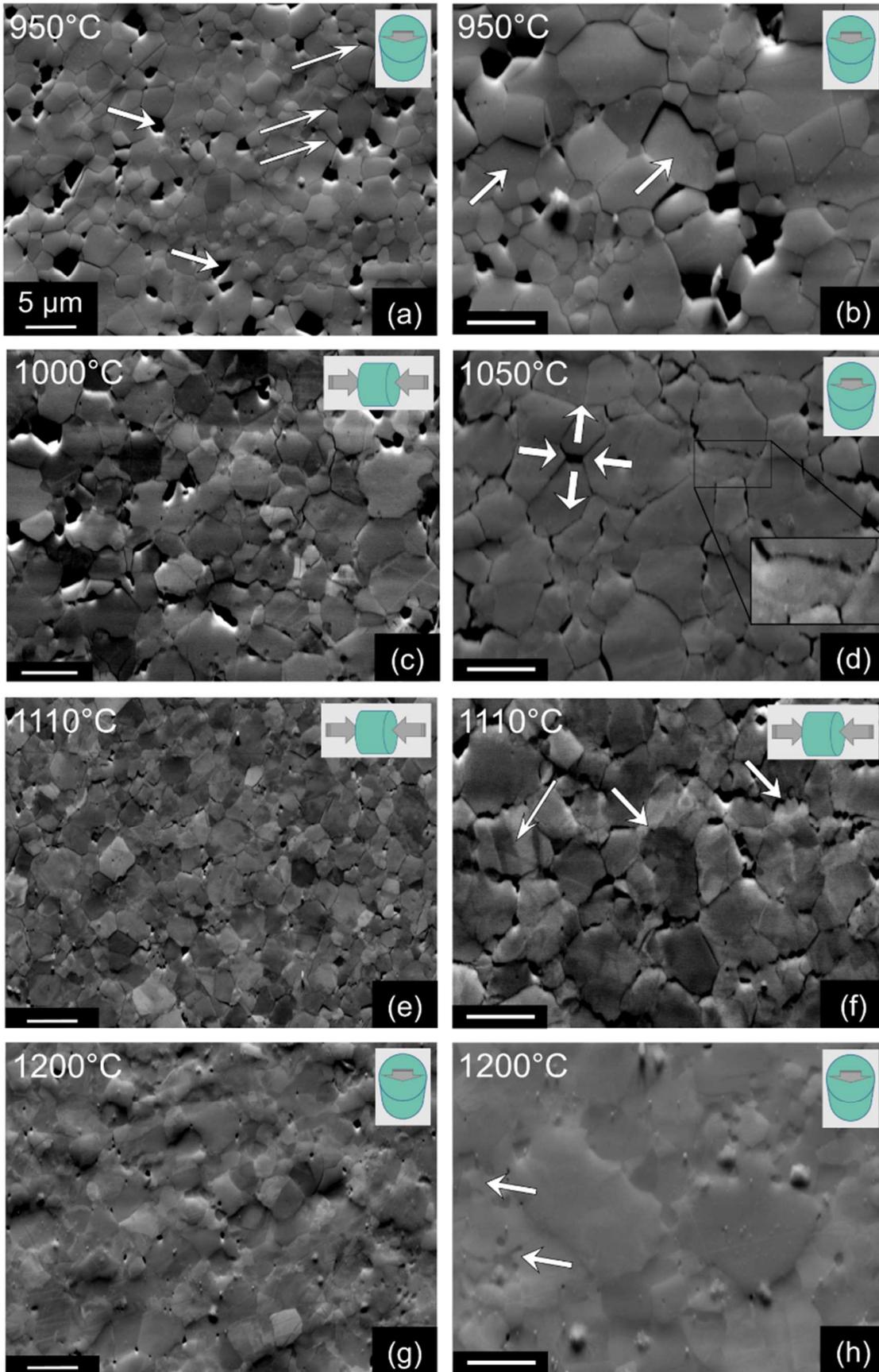
Figure 4: SEM images (forward scattered electrons) of samples deformed at various temperatures. Scale bar is 5 μm on all images. The direction of the deformation (either horizontal or in-plane) is given by the

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.

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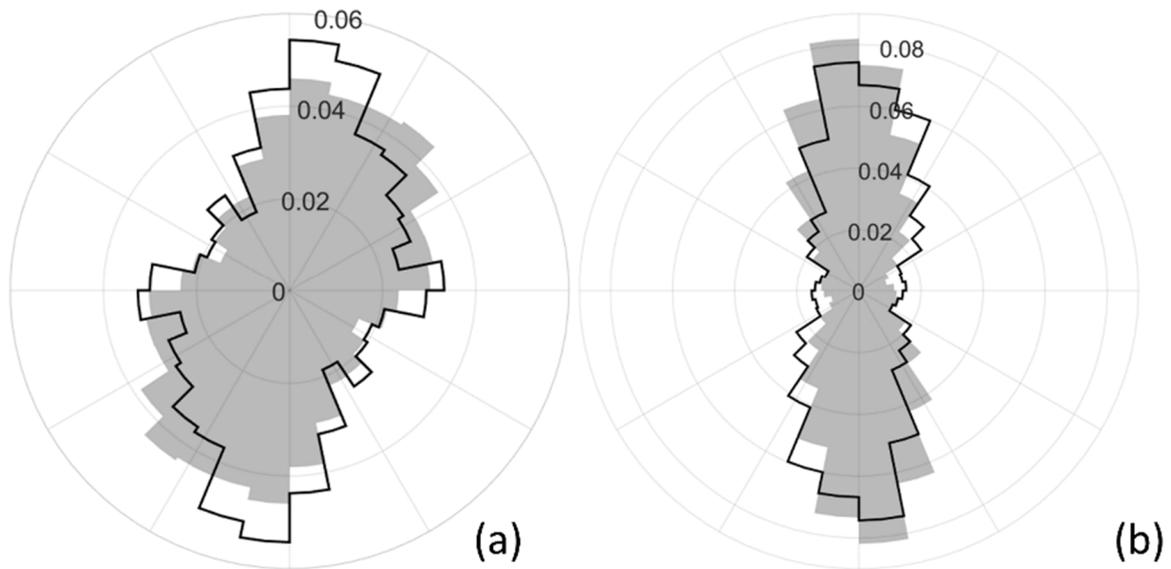
Figure 5: Polar histograms displaying the normalized orientation distribution of the longest axis of forsterite grains. Samples length, and therefore axial compression direction, is horizontal. (a) Starting material (black line) and sample NF_900-1 (grey bars), deformed to a finite strain of 3.9 %. (b) Samples NF_1110-2 (black line) and NF_1110-1 (grey bars) deformed by 5 and 12.41 % of finite strain, respectively.
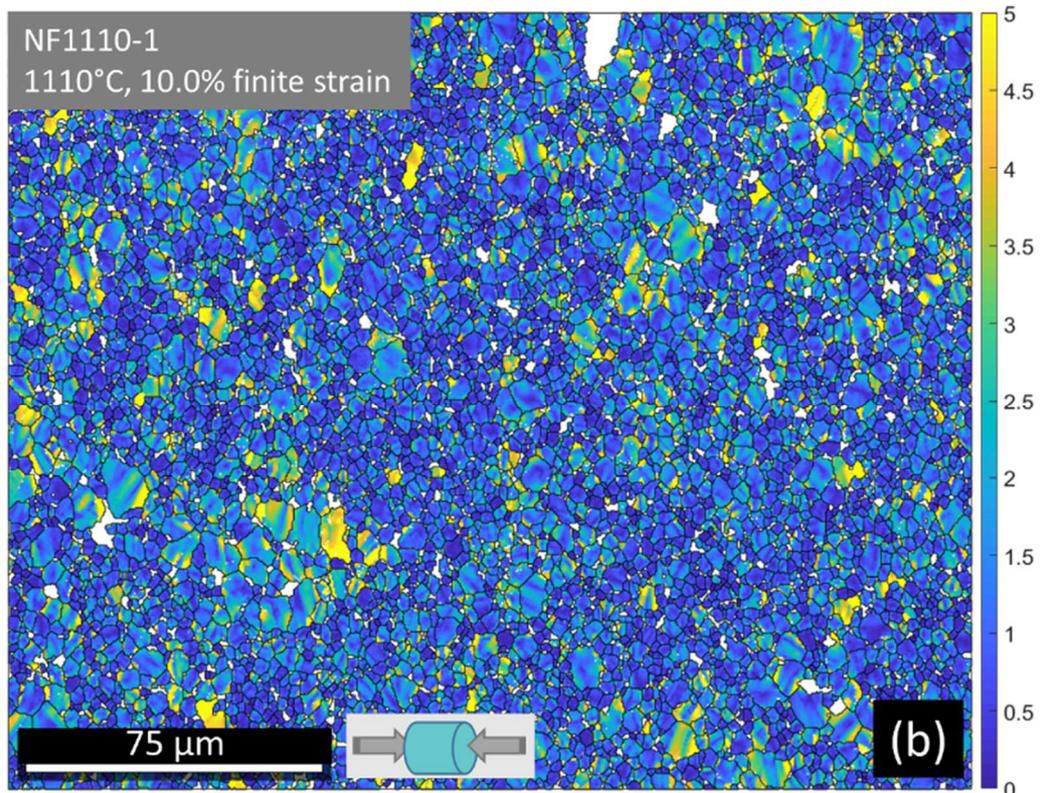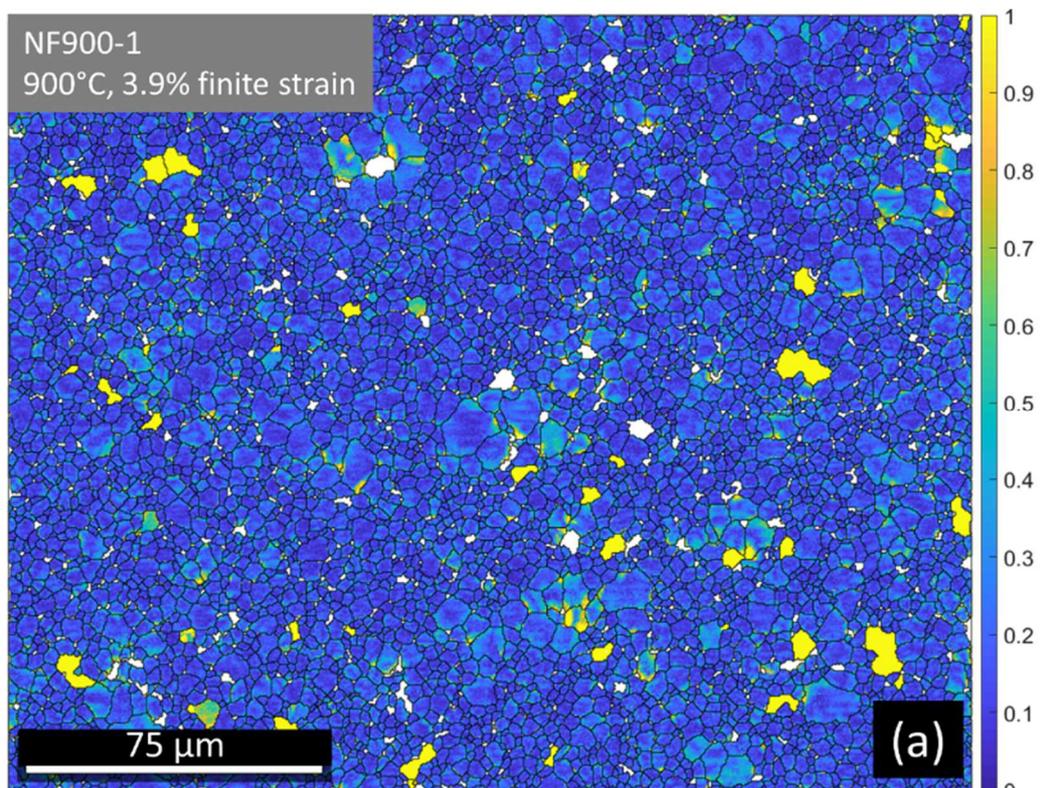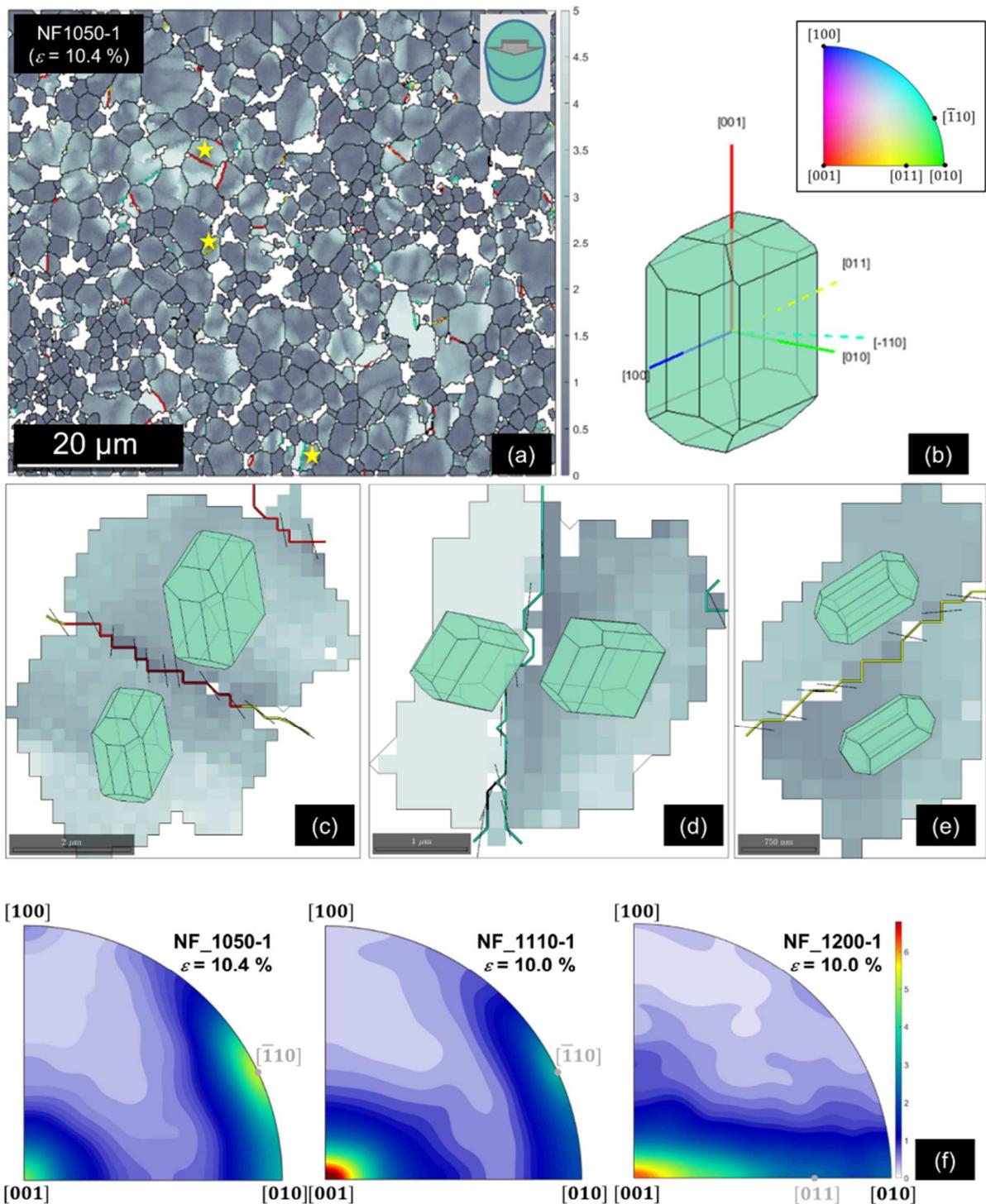
Figure 6: EBSD maps showing grain boundaries and Mis2Mean angles (°) within each grain. (a) Sample NF_900-1 (3.9 % finite strain). Very limited grain plasticity is observed, as shown by Mis2Mean values, which rarely exceed 1°. (b) Sample NF_1110-1 (12.41 % finite strain), which presents the largest GOS values observed in our samples. Note that the color scale maximum in (b) is five times larger than in (a).
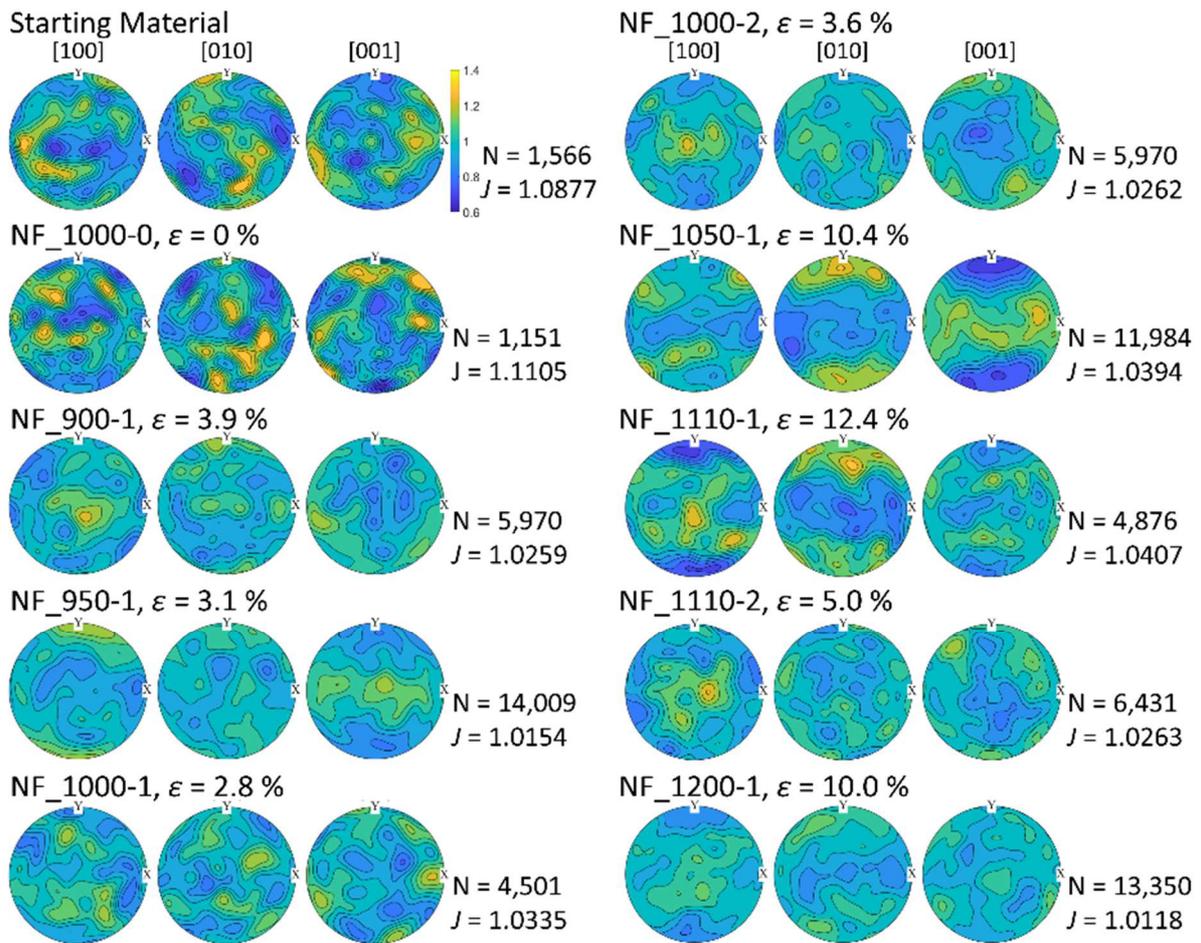
Figure 7: Pole figures of forsterite obtained for the starting material, the annealing experiment and each axial deformation experiment. Densities were obtained from the largest EBSD map collected on each sample and reduced to one point per grain. Densities were contoured with a 0.06 interval. The color scale given beside the starting material pole figures is the same on all figures. The number of grains used, N, and the corresponding J-index are provided in each case. The direction of axial compression deformation is vertical for all figures, except for sample NF_900-1, where orientation was lost due brittle failure of the sample.

Figure 8: Intracrystalline plasticity at 1050-1200 °C. (a)-(e) Misorientation analysis in sample NF_1050-1. (a) EBSD map showing Mis2Mean values, grain boundaries (black lines) and subgrain boundaries (thick colored lines). (b) Reference forsterite crystal habitus used in (c), (d) and (e). In addition to principal axes, specific axes, corresponding to $[\bar{1}10]$ and [011] subgrain rotations, are shown and colored according to the inverse pole figure colors given in the inset. (c)-(e) Detailed views of grains (and subgrains) marked by a star in (a). The bars on top of the subgrain boundaries represent the orientation of the subgrain rotation axis; their 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.
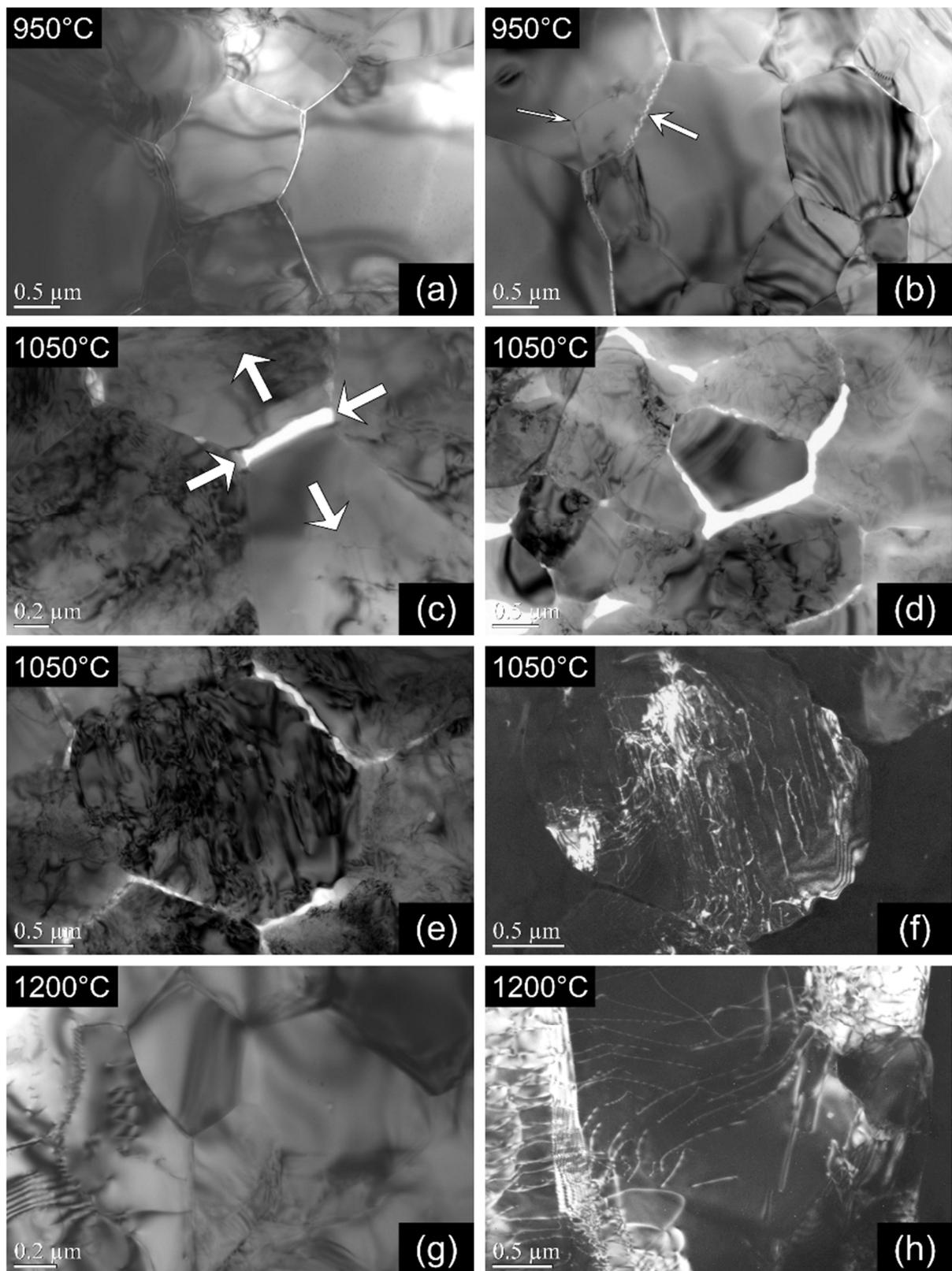
Figure 9: TEM images of samples deformed at 950-1200 °C. (a)-(b) Sample NF_950-1. (a) Grains show few to no dislocation. Gaping grain boundaries are straight, typical of brittle failure. (b) Several grain boundaries are serrated at the scale of a few tens of nanometers (thick arrow) and intragranular cracks are also present (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.
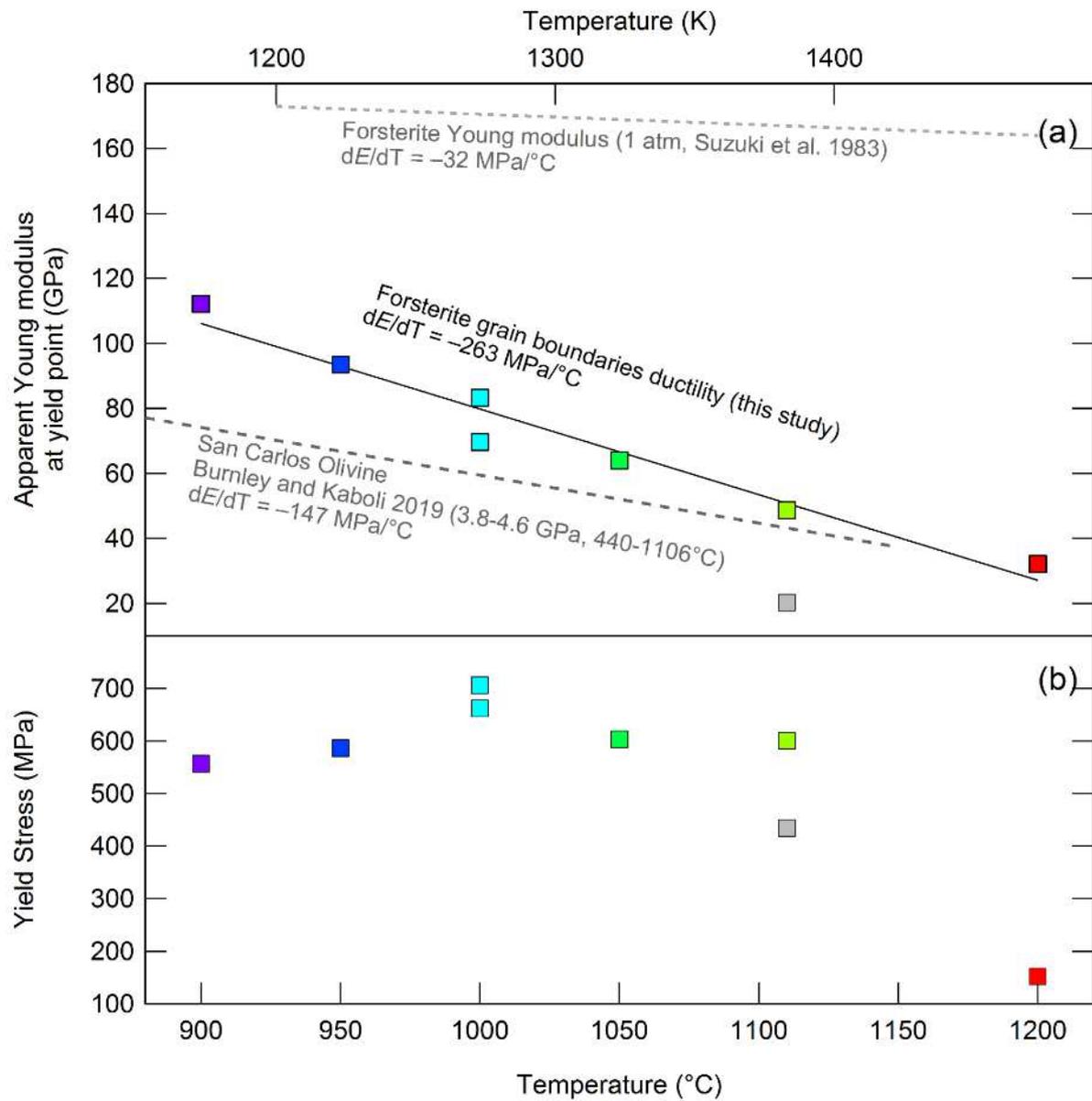
Figure 10: (a) Apparent Young modulus at yield point as a function of temperature. Young modulus of forsterite as a function of temperature from Suzuki et al. (1983) is shown for comparison. Values of grain boundaries apparent elasticity for iron-bearing olivine from Burnley and Kaboli (2019) are also reported. (b) Yield stress as a function of temperature from the mechanical curves displayed in Figure 2b (see Supp Mat for details, Figure S2).
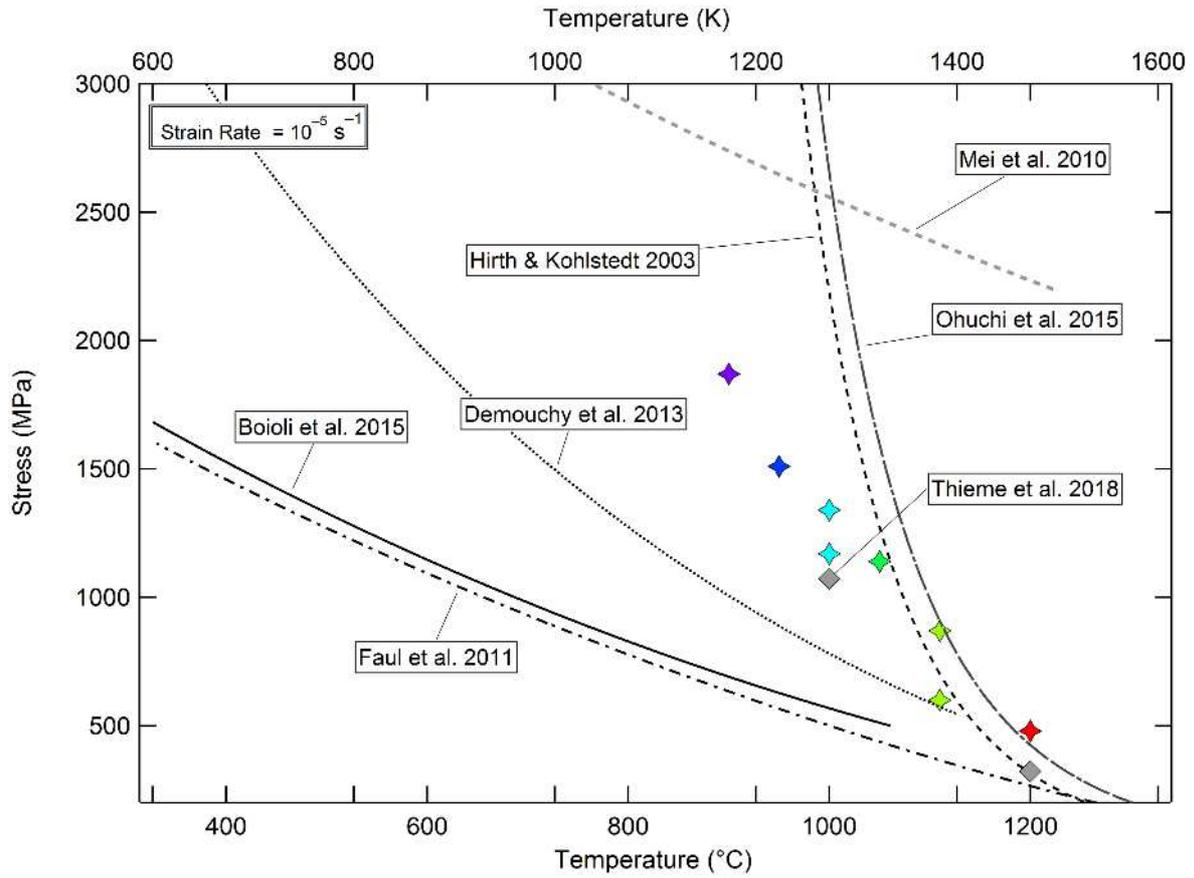
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807   Figure 11: Comparison of iron-bearing olivine strength from the literature with the mechanical data from

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 E 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
	J 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	1.082	1.0449	1.0432	1.0235	1.3568

_J_ index 1PPG	1.0877	1.1285	1.1105	1.1335	1.0314	1.0259	1.0949	1.0154	1.0335	1.0364	1.0262	1.0857	1.0394	1.2434	1.0407	1.0453	1.0263	1.0246	1.0118	1.1569
Average Grain Size (µm)	3.115		3.285		3.085		2.660		3.250		2.780		2.925		3.120		2.725		2.860	
Mean Aspect Ratio	1.925		1.930		1.845		2.055		1.920		1.910		2.085		2.125		1.940		2.165	
Mean Shape Factor	1.390		1.385		1.390		1.430		1.425		1.400		1.500		1.440		1.395		1.485	
Mean GOS (°)	0.210		0.200		0.245		0.340		0.375		0.260		1.310		1.375		0.565		0.705	