

# 1 Frictional properties and microstructural evolution of dry and wet 2 calcite-dolomite gouges

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## 14 Key Points

- 15 • Presence of water during deformation has a significant effect on the frictional and  
16 microstructural evolution of calcite-dolomite gouges
- 17 • Gouge fluidization is observed at slip rates of  $30 \mu\text{ms}^{-1}$  –  $0.1 \text{ms}^{-1}$  under water-  
18 dampened conditions
- 19 • Development of gouge foliation is observed only at coseismic slip rates and room-  
20 humidity conditions

21

## 22 Keywords

23 Carbonates, earthquake, rock deformation, gouge, calcite, dolomite, friction, microstructure

24

## 25 Abstract

26 Calcite and dolomite are the two most common minerals in carbonate-bearing faults  
27 and shear zones. Motivated by [observations of](#) exhumed seismogenic faults in the Italian  
28 Central Apennines, we [used a rotary-shear apparatus to investigate](#) the frictional and  
29 microstructural evolution of [c. 3 mm-thick](#) gouge [layers](#) consisting of 50 wt.% calcite and 50  
30 wt.% dolomite. The gouges were sheared at a range of slip rates ( $30 \mu\text{ms}^{-1}$  –  $1 \text{ms}^{-1}$ ),  
31 displacements (0.05–0.4 m), and normal [load of 17.5](#), under both room-humidity and water-  
32 dampened conditions. The frictional behaviour and microstructural evolution of the gouges  
33 were strongly influenced by the presence of water. At room humidity, slip strengthening was  
34 observed up to slip rates of  $0.01 \text{ms}^{-1}$ , which was associated with gouge dilation and the  
35 development of a 500-900  $\mu\text{m}$  wide slip zone cut by  $Y_1$ ,  $R_1$ , and  $R_2$ -shear bands. Above a slip

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45 rate of  $0.1 \text{ ms}^{-1}$ , dynamic weakening accompanied the development of a localised  $<100 \mu\text{m}$   
 46 thick principal slip zone preserving microstructural evidence for calcite recrystallization and  
 47 dolomite decarbonation, while the bulk gouges developed a well-defined foliation consisting of  
 48 organized domains of heavily fractured calcite and dolomite. In water-dampened conditions,  
 49 evidence of gouge fluidization within a fine-grained principal slip zone was observed at a range  
 50 of slip rates from  $30 \mu\text{ms}^{-1}$  to  $0.1 \text{ ms}^{-1}$ , suggesting that caution is needed when relating  
 51 fluidization textures to seismic slip in natural fault zones. Dynamic weakening in water-  
 52 dampened conditions was observed at  $1 \text{ ms}^{-1}$ , where the principal slip zone was characterised  
 53 by patches of recrystallized calcite. However, local fragmentation and reworking of  
 54 recrystallized calcite suggests a cyclic process involving formation and destruction of a  
 55 heterogeneous slip zone. Our microstructural data show that development of well-defined  
 56 gouge foliation under the tested experimental conditions is limited to high-velocity ( $>0.1 \text{ ms}^{-1}$ )  
 57 and room humidity, supporting the notion that some foliated gouges and cataclasites may form  
 58 during seismic slip in natural carbonate-bearing faults.

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## 60 1. Introduction

61 Calcite and dolomite are the most common minerals in carbonate-bearing faults and  
 62 shear zones (e.g. Busch and Van Der Pluijm, 1995; Snoke et al., 1998; Bestmann et al., 2000;  
 63 De Paola et al., 2006; Molli et al., 2010; Tesei et al., 2014; Fondriest et al., 2015, 2020; Delle  
 64 Piane et al., 2017). In some cases, the distribution and timing of dolomitization plays an  
 65 important role in controlling strain localization. For example, ductile deformation along the  
 66 Naukluft Nappe Complex in central Namibia was distributed within a sequence of calcite  
 67 mylonites, but the main Naukluft Fault localized within dolomitized layers (Viola et al., 2006;  
 68 Miller et al., 2008).

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69 Although similar in composition, the rheology, deformation mechanisms, and frictional  
 70 behaviour of calcite and dolomite show important differences. Deformation of calcite has been  
 71 widely investigated using microstructural analysis (Kennedy and Logan, 1997; Kennedy and  
 72 White, 2001; Liu et al., 2002; Bestmann et al., 2006; Molli et al., 2011) and laboratory  
 73 experiments over a wide range of conditions, which includes experiments performed at  
 74 relatively low strain rates ( $<10^{-2} \text{ s}^{-1}$ ), high temperatures ( $>500 \text{ }^\circ\text{C}$ ), and high pressures ( $>100$   
 75 MPa) (e.g. Rutter, 1972; Schmid et al., 1980, 1987; de Bresser et al., 1990; Rutter, 1995;  
 76 Paterson and Olgaard, 2000), as well as experiments performed at relatively high shear rates  
 77 ( $>1 \mu\text{ms}^{-1}$ ), low temperatures ( $<150 \text{ }^\circ\text{C}$ ), and low pressures ( $<50 \text{ MPa}$ ) (Smith et al., 2013;  
 78 Verberne et al., 2014; De Paola et al., 2015; Smith et al., 2015; Rempe et al., 2017; Tesei et  
 79 al., 2017). Comparatively, the rheology and frictional behaviour of dolomite is relatively poorly

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100 understood (e.g., Barber et al., 1981; Weeks and Tullis, 1985). Recently, the importance of  
101 dolomite as a fault and shear zone material in sedimentary and metamorphic settings has been  
102 emphasized in a number of experimental studies (Austin and Kennedy, 2005; Delle Piane et  
103 al., 2007, 2008; Davis et al., 2008; De Paola et al., 2011a, 2011b; Boneh et al., 2013; Fondriest  
104 et al., 2013; Holyoke et al., 2014; Green et al., 2015). At low strain rates, dolomite is brittle up  
105 to c. 700°C (Kushnir et al., 2015), while calcite can undergo recrystallization at temperatures  
106 as low as 150–200°C (Kennedy and White, 2001). This pronounced difference in deformation  
107 style under similar ambient conditions may significantly influence the rheology of faults and  
108 shear zones in which the two phases co-exist (Oesterling et al., 2007; Kushnir et al., 2015).

109 Only a few experimental studies have investigated the mechanical behaviour and  
110 microstructural evolution of calcite-dolomite mixtures. Experiments have been performed to  
111 study the rheological behaviour of mixtures at relatively high pressures and temperatures (e.g.,  
112 torsion experiments of Delle Piane et al., 2009; Kushnir et al., 2015), as well as the frictional  
113 behaviour of mixtures at room temperature over a wide range of strain rates (e.g., room  
114 temperature rotary-shear experiments: Mitchell et al., 2015; Smith et al., 2017; Demurtas et  
115 al., 2019a,b). In torsion experiments (confining pressures up to 300 MPa, temperatures of 700–  
116 800 °C, shear strain rate  $\dot{\gamma} = 1\text{--}3 \times 10^{-4} \text{ s}^{-1}$ , and finite shear strain  $\gamma < 11$ ), minor quantities of  
117 dolomite (e.g., 25 wt.%) in a sintered calcite-rich sample significantly increased the yield  
118 strength with respect to pure calcite samples (Kushnir et al., 2015). Under such experimental  
119 conditions, two main deformation mechanisms were observed: brittle fracturing in the dolomite  
120 grains and ductile flow in calcite, possibly as a result of grain boundary sliding assisted by  
121 diffusion creep and dislocation glide (Kushnir et al., 2015). Strain hardening observed in these  
122 experiments was interpreted to be due to dolomite grains interrupting more continuous calcite-  
123 rich layers and acting as stress concentrators. Brittle failure of dolomite grains eventually  
124 allowed the calcite-rich layers to become continuous and to continue deforming by superplastic  
125 flow (Kushnir et al., 2015). Although these torsion experiments were performed under  
126 significantly elevated temperatures and pressures compared to the experiments in this paper,  
127 the results suggest that the occurrence of dolomite in calcite aggregates can influence the  
128 mechanical behaviour during deformation.

129 Mitchell et al. (2015) and Smith et al. (2017) studied the frictional behaviour and  
130 microstructural evolution of gouge mixtures (50 wt.% dolomite and 50 wt.% calcite) deformed  
131 at low normal stresses ( $\sigma_n \leq 17.5 \text{ MPa}$ ), high slip rates ( $V \geq 0.01 \text{ ms}^{-1}$ ), and large displacements  
132 ( $d = 0.03\text{--}3 \text{ m}$ ), with the aim of reproducing conditions encountered at the base of fast-moving  
133 landslides and during the seismic cycle in shallow-crustal faults. At a slip velocity of  $1 \text{ ms}^{-1}$ ,  
134 dynamic weakening was associated with grain size reduction and decarbonation of dolomite

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143 within the experimental principal slip zone and in the nearby bulk gouge. During the early  
144 stages of these high-velocity experiments, and prior to the onset of dynamic weakening, a well-  
145 defined foliation developed within the [bulk](#) gouge mixtures due to brittle fracturing of calcite  
146 and dolomite accompanied by shearing of the fractured grains in to compositional bands  
147 (Mitchell et al., 2015; Smith et al., 2017). These observations indicate that some natural  
148 examples of foliated gouges and cataclasites could form during coseismic shearing (Smith et  
149 al., 2017), challenging the common interpretation that fault rock foliations result from slow  
150 aseismic creep (e.g., Rutter et al., 1986; Chester and Chester, 1998; Lin, 2001; Jefferies et al.,  
151 2006). Additionally, Demurtas et al. (2019a) documented the presence of a well-defined  
152 crystallographic preferred orientation (CPO) in calcite-dolomite gouges, and interpreted the  
153 CPO to result from “brittle” processes involving grain rotation and preferential fracturing along  
154 calcite cleavage planes during granular flow at room temperature. Instead, in regions of the  
155 mixed gouge layers that experienced substantial frictional heating during high-velocity slip (i.e.,  
156 within the principal slip zone), Transmission Kikuchi Diffraction analysis suggested that  
157 nanogranular aggregates deformed by a combination of grain size sensitive (grains <800 nm)  
158 and grain size insensitive (grains >800 nm) plastic creep.

159 The experimental work summarized above indicates that a diverse range of  
160 microstructures can form in calcite-dolomite gouges as a result of both brittle and plastic  
161 processes, and that the prevailing microstructures depend on ambient conditions, strain  
162 history, and proximity to zones of shear localization and heating. Potentially, this range of  
163 microstructures could be recognized in natural gouges and cataclasites, which would provide  
164 important insights in to the evolution of slip conditions during the seismic cycle in carbonate-  
165 bearing faults. However, to successfully apply the experimental findings to natural fault zones,  
166 a more complete picture of microstructural diversity and its dependence on deformation  
167 conditions is required. In this context, the aim of this paper is to provide (i) a more  
168 comprehensive description of the frictional and microstructural evolution of mixed calcite-  
169 dolomite gouges deformed at sub-seismic to seismic slip rates, and (ii) an updated framework  
170 for the interpretation of microstructures found in natural calcite- and dolomite-bearing faults.

171

## 172 **2. Methods**

### 173 **2.1. Starting materials**

174 Synthetic gouges were prepared by mixing 50 wt.% calcite and 50 wt.% dolomite as  
175 previously described in Demurtas et al. (2019a). The calcite-dolomite ratio in the experimental  
176 mixtures is similar to that found in natural fault gouges and cataclasites from the Vado di Corno  
177 Fault Zone (VCFZ, Italian Central Apennines; Demurtas et al., 2016), and used in previous

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180 experimental studies (Smith et al., 2017). The calcite gouge was derived by crushing Carrara  
181 marble with a modal composition of 98.8 wt.% calcite and <1 wt.% dolomite and muscovite  
182 (see Supplementary Material). The dolomite gouge was derived by crushing dolomitized  
183 portions of the Calcare Massiccio Formation from the VCFZ (Demurtas et al., 2016). The  
184 crushed gouges were passed through a 250  $\mu\text{m}$  sieve and then mixed together by slow  
185 tumbling for c. 30 minutes. Two batches of gouge were prepared (CDM1, calcite = 47.2 wt.%  
186 and dolomite = 52.8 wt.%; CDM2, calcite = 42.9 wt.% and dolomite = 57.1 wt.%).

187

## 188 **2.2. Experimental setup and deformation conditions**

189 Nineteen experiments were performed at slip rates from 30  $\mu\text{ms}^{-1}$  to 1  $\text{ms}^{-1}$  with SHIVA  
190 (Slow- to High-Velocity rotary-shear friction Apparatus) at the Istituto Nazionale di Geofisica e  
191 Vulcanologia in Rome (Di Toro et al., 2010; Niemeijer et al., 2011) (Table 1). The gouges were  
192 deformed inside a metal holder specifically designed for incohesive materials (Fig. 1; Smith et  
193 al., 2013, 2015). The thickness of the gouge layers at the start of the experiments was c. 3  
194 mm. Horizontal displacements of the axial column were sampled at 2.5 Hz–25 kHz, and  
195 measured using a direct current differential transformer (DCDT, 50 mm range and c. 50  $\mu\text{m}$   
196 resolution) and a linear variable differential transformer (LVDT, 3 mm range and c. 0.03  $\mu\text{m}$   
197 resolution). Further details of the data acquisition system, and location and calibration of the  
198 load cells, detectors, and devices, are found in Niemeijer et al. (2011) and Smith et al. (2013).

199 Measurements of room humidity and room temperature were collected at a distance of  
200 <1 cm from the gouge holder before and during the experiments (Fig. 1a). Temperature  
201 variations during deformation were measured at an acquisition rate of 2.5 Hz using four K-type  
202 thermocouples (Nickel-Alumel) installed on the stationary side of the gouge holder (Fig. 1a-b;  
203 Demurtas et al., 2019a). One thermocouple was positioned at c. 200  $\mu\text{m}$  from the gouge layer  
204 (Fig. 1b). The other three thermocouples were located in the sample holder and stationary  
205 column to detect temperature variations due to heat conduction through the gouge holder and  
206 apparatus (Fig. 1a).  $\text{CO}_2$  emissions were monitored using an OmniStar™ GSD 301 O mass  
207 spectrometer designed for gas analysis at atmospheric pressure.

208 Experiments were performed under both room-humidity and water-dampened  
209 conditions at a constant normal stress of  $17.5 \pm 0.1$  MPa (Table 1), the same normal stress  
210 used in some previous SHIVA experiments conducted on calcite, dolomite, and calcite-  
211 dolomite gouges (Fondriest et al., 2013; Smith et al., 2015, 2017). Room humidity varied  
212 between 41% and 62%, and room temperature between 19 °C and 22 °C. In water-dampened  
213 conditions, c. 2 ml of deionized water was added to the top of the gouge layer using a pipette  
214 before the gouge holder was positioned in the apparatus. Experiment s1327 was performed

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223 using a specially designed water bath that ensured saturation within the gouge layer during  
224 this long-duration experiment (Supplementary Material). [The gouge holder used in the](#)  
225 [experiments did not allow us to measure pore-fluid pressure during water-dampened](#)  
226 [experiments](#). Experiments were performed at target slip rates ranging from  $30 \mu\text{ms}^{-1}$  to  $1 \text{ms}^{-1}$ ,  
227 with acceleration and deceleration of  $6 \text{ms}^{-2}$ . Total displacements ranged from 0.05 m to 0.4  
228 m. Two compaction experiments were performed by applying a normal stress of 17.5 MPa for  
229 300 s (i.e., static load experiments in Table 1) and used as references for the microstructure  
230 of the starting materials.

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### 231 232 **2.3 Analytical techniques**

233 After each experiment, the entire gouge layer was recovered and impregnated in low-  
234 viscosity epoxy (Araldite 2020) for microstructural analysis. Polished thin sections were cut  
235 perpendicular to the gouge layer and subparallel (i.e., tangential cut) to the slip direction (Fig.  
236 1d). Microstructural analysis was performed with a Zeiss Sigma VP Field-Emission Scanning  
237 Electron Microscope (SEM) at the Otago Micro and Nanoscale Imaging facility (OMNI;  
238 University of Otago. Acquisition conditions for backscattered electron images: accelerating  
239 voltage 15 kV, working distance 6-7 mm). Energy-dispersive X-ray spectroscopy (EDS) in the  
240 SEM was used to produce element maps showing the distribution of calcium and magnesium,  
241 [which highlights the distribution of calcite and dolomite](#). Crystallographic orientation data from  
242 calcite were acquired by electron backscatter diffraction (EBSD) on SYTON-polished thin  
243 sections. Data were collected with a NordlysF EBSD camera from Oxford Instruments and  
244 processed using AZtec software (Oxford Instruments). Mineralogical changes that occurred  
245 during the experiments were determined by semi-quantitative X-ray powder diffraction (XRPD)  
246 conducted in the Department of Geoscience, University of Padova. The XRPD analyses were  
247 performed on both the bulk gouges and on small intact chips of the localized slip surfaces that  
248 formed in the experiments.

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## 249 250 **3. Results**

### 251 **3.1. Friction evolution with slip and slip rate**

252 The evolution of the effective friction coefficient ( $\mu$ ) with slip and slip rate was influenced  
253 by the availability of water during deformation (Figs. 2, 3). In room-humidity conditions and slip  
254 rates of  $\leq 0.01 \text{ms}^{-1}$ , the calcite-dolomite mixtures showed a progressive increase of  $\mu$  (slip  
255 strengthening behaviour) up to 0.75-0.80 (measured between 0.15 m and 0.35 m of slip)  
256 following an initial peak friction ( $\mu_{\text{peak}}$ ) of 0.64-0.71 (Figs. 2, 3). At a slip rate of  $0.1 \text{ms}^{-1}$ , a  
257 substantial decrease of  $\mu$  was observed (slip weakening behaviour to steady state  $\mu_{\text{ss}}$  of

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262 0.55±0.01) following a prolonged initial strengthening phase (c. 0.062 m) that reached  $\mu_{peak}$  of  
263 0.68 (Fig. 2b). Significant dynamic weakening was observed at a slip rate of 1 ms<sup>-1</sup> following  
264 a short initial strengthening phase (lasting c. 0.005 m) that was followed by steady state  $\mu_{ss}$  of  
265 0.28 (Figs. 2a, 3b). A re-strengthening phase (final  $\mu$  up to c. 0.56) was observed during  
266 deceleration of the rotary column.

267 In water-dampened conditions, the gouge mixtures showed a similar evolution of  
268 friction at slip rates ≤ 0.1 ms<sup>-1</sup>, characterized by slight slip strengthening to slip neutral  
269 behaviour (Fig. 2c). Notably,  $\mu_{peak}$  and  $\mu_{ss}$  were lower than in room-humidity experiments, with  
270  $\mu_{peak}$  = 0.61-0.64 and  $\mu_{ss}$  = 0.62-0.70 (Fig. 3). At a slip rate of 1 ms<sup>-1</sup>, the initial strengthening  
271 phase was much shorter than in room-humidity conditions (c. 0.003 m), and dynamic  
272 weakening resulted in  $\mu_{ss}$  of 0.31±0.02. Re-strengthening was also observed during  
273 deceleration, with an increase in  $\mu$  up to 0.57.

274

### 275 3.2. Gouge thickness evolution with slip rate

276 No significant gouge loss was observed during the experiments, with the exception of  
277 those performed at V = 0.1 ms<sup>-1</sup> discussed below. Therefore, the evolution of axial  
278 displacement is interpreted to result from changes in gouge layer thickness due to dilation and  
279 compaction. In room-humidity conditions, the evolution of gouge layer thickness depends on  
280 slip rate (Fig. 4a). At V ≤ 0.001 ms<sup>-1</sup>, the gouge layers show a three-stage evolution: (i) initial  
281 compaction of c. 90-120 μm at the onset of sliding, (ii) dilation of c. 50-70 μm during the slip  
282 strengthening phase, and (iii) approximately constant thickness once the steady state friction  
283 coefficient is reached. Overall compaction of c. 30-60 μm is recorded. At V = 0.01 ms<sup>-1</sup>, after  
284 a short lived dilatancy phase (inset in Fig. 4a), compaction of 100 μm is followed by  
285 approximately constant thickness (Fig. 4a). At higher slip rates (V ≥ 0.1 ms<sup>-1</sup>), continuous  
286 compaction was observed throughout the experiments (up to c. 300 μm of axial shortening at  
287 V = 1 ms<sup>-1</sup>), and compaction rate increased with slip rate (Fig. 4a).

288 Under water-dampened conditions, the gouge mixtures exhibit a similar evolution of  
289 thickness irrespective of slip rate (Fig. 4b). Compaction was initially rapid in the first few cm of  
290 sliding, and then reached an approximately constant compaction rate that was similar in all  
291 experiments. Total compaction of c. 200-250 μm was recorded (Fig. 4b).

292

### 293 3.3. Temperature evolution and CO<sub>2</sub> emissions

294 Figure 5a shows maximum temperatures measured by the thermocouple located  
295 closest to the gouge layers (Fig. 1b; Demurtas et al., 2019a described temperature evolution  
296 with slip). The maximum temperature (621 °C) was achieved in experiment s1221 performed

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309 under room-humidity conditions at  $V = 1 \text{ ms}^{-1}$  (Fig. 5a). For the same slip rate and normal  
310 stress, but in water-dampened conditions, the maximum temperature was  $210 \text{ }^\circ\text{C}$  (Fig. 5a).  
311 Temperature increases were detected in all experiments at slip rates  $\geq 0.01 \text{ ms}^{-1}$ , and the  
312 maximum temperature increased with increasing slip rate (Fig. 5a).

313 [Because the OmniStar mass spectrometer acquisition system used to detect CO<sub>2</sub>](#)  
314 [emissions is independent from the control system on SHIVA, CO<sub>2</sub> emissions are plotted simply](#)  
315 [against the time \(in seconds\) at which the mass spectrometer was started, which is variable](#)  
316 [for each experiment \(Fig. 5b\).](#) CO<sub>2</sub> emissions above ambient levels were only detected in  
317 experiments at slip rates  $\geq 0.1 \text{ ms}^{-1}$  (Fig. 5b). Because the mass spectrometer was not  
318 calibrated and the sample holder was open to the laboratory, the data can only be used in a  
319 qualitative way. In room-humidity conditions, the intensity of the CO<sub>2</sub> peak was significantly  
320 higher at  $1 \text{ ms}^{-1}$  than at  $0.1 \text{ ms}^{-1}$ . In water-dampened conditions, the CO<sub>2</sub> peaks were  
321 substantially smaller than at equivalent room-humidity conditions.

### 323 3.4. Mineralogy of deformed gouges

324 Compared to the starting materials, no mineralogical changes were detected in any of  
325 the deformed bulk gouges (see Supplementary Material). In room-humidity experiment *s1210*  
326 ( $30 \mu\text{ms}^{-1}$ ), a slight broadening of the main peak for calcite was observed (Fig. 6a), and to a  
327 lesser degree also for dolomite. XRPD analysis of cohesive chips recovered from the slip  
328 surface of water-dampened experiment *s1214* ( $V = 30 \mu\text{ms}^{-1}$ ) indicates the presence of  
329 aragonite (Fig. 6b). At  $V = 1 \text{ ms}^{-1}$  and room-humidity conditions, (experiment *s1221*), the  
330 recovered slip surface was composed of dolomite, Mg-calcite, and periclase (MgO) (Fig. 6c).  
331 Mg-calcite and periclase are two of the main products of dolomite decarbonation that starts at  
332 c.  $550 \text{ }^\circ\text{C}$  ( $\text{MgCa}(\text{CO}_3)_2 \Rightarrow \text{MgO} + (\text{Ca}, \text{Mg})\text{CO}_3 + \text{CO}_2$ , Samtani et al., 2002; De Paola et al.,  
333 2011a,b).

### 335 3.5. Microstructures of deformed gouge layers

#### 336 3.5.1. Microstructures of room-humidity experiments

337 At slip rates  $\leq 0.01 \text{ ms}^{-1}$ , gouges were characterized by the development of a 500-900  
338  $\mu\text{m}$  thick slip zone (Figs. 7a and 8), consisting of a fine-grained matrix (grain size c.  $1 \mu\text{m}$ )  
339 containing subrounded grains of dolomite c. 5-10  $\mu\text{m}$  in size (Fig. 7b). The slip zone contains  
340 sub-parallel, 10-30  $\mu\text{m}$  thick Y, R, and R<sub>1</sub> type shear bands (using the terminology of Logan et  
341 al., 1979; Fig. 7a,c). Each individual shear band is associated with a very fine-grained matrix  
342 (grain size  $< 1 \mu\text{m}$ ) composed of calcite and dolomite. The presence of multiple interlinked  
343 shear bands contributes to a weak foliation within the slip zone that lies sub-parallel to gouge

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367 layer boundaries (Fig. 7a). Y-, R-, and most notably R<sub>1</sub>-shears, gradually decrease in  
368 abundance with increasing slip rate. The transition from fine-grained slip zone to highly  
369 fractured bulk gouge is typically well-defined (see upper part in Fig. 7d). The bulk gouge shows  
370 widespread cataclasis and intragranular fracturing, which is focussed preferentially into calcite  
371 grains (Smith et al., 2017; Demurtas et al., 2019a). Fractures that cut relatively large grains of  
372 calcite in the bulk gouge often exploit cleavage planes (e.g., Fig. 7d; Smith et al., 2017;  
373 Demurtas et al., 2019a).

374 At a slip rate of 0.1 ms<sup>-1</sup>, the bulk gouge develops a weak foliation defined by  
375 compositional banding of heavily fractured calcite- and dolomite-rich domains, which lie  
376 adjacent to a localized principal slip zone c. 110 μm thick (Fig. 7e). The foliation is inclined 25-  
377 30° to the principal slip surface and appears to form by disaggregation and shearing of  
378 originally intact calcite and dolomite grains (Fig. 7e). Locally, the principal slip surface is  
379 associated with discontinuous lens-shaped patches (up to 15-20 μm thick) of calcite with  
380 irregular boundaries and negligible porosity (Fig. 7f).

381 In experiments conducted at 1 ms<sup>-1</sup>, the bulk gouges developed a well-defined foliation  
382 across most of the thickness of the layers (Fig. 9; Smith et al. 2017; Demurtas et al. 2019a,b)  
383 The foliation is defined by alternating calcite- and dolomite-rich domains inclined at c. 40° to  
384 the principal slip surface (Fig. 9a-b), which become progressively rotated as they approach the  
385 slip surface (Fig. 9c). Large remnant grains (up to 200 μm) in the bulk gouge are often rimmed  
386 by fractured tails of finer-grained aggregates (grain size <10 μm), and resemble mantled  
387 porphyroclasts in mylonites (e.g., Snoke et al., 1998; Trouw et al., 2009) (arrow in Fig. 9a). At  
388 distances of <400 μm from the principal slip surface, the mean grain size decreases  
389 substantially, there are very few large surviving grains (up to c. 100 μm in size), and there is a  
390 greater degree of mixing between calcite and dolomite (see more uniform colouring in the  
391 upper part of EDS map in Fig. 9b). The principal slip zone consists of a 15-20 μm thick,  
392 extremely fine-grained layer (<<1 μm in size) composed of calcite, Mg-calcite, dolomite, and  
393 periclase (EDS and XRPD analysis; Figs. 6 and 9c-d). Calcite forms elongate aggregates with  
394 negligible porosity that display an aggregate preferred orientation with the long axes sub-  
395 parallel to foliation (Fig. 9c-e). Dolomite-rich domains show higher porosity and preserve  
396 distinct grain structures (Fig. 9c-d). EBSD analysis of elongate calcite aggregates adjacent to  
397 the principal slip zone (Fig. 9e) shows a distinct crystallographic preferred orientation with c-  
398 axes inclined sub-perpendicular to gouge layer boundaries (Fig. 9f; see also Demurtas et al.,  
399 2019b). Locally, a c. 30-40 μm thick layer adjacent to the principal slip zone, includes dolomite  
400 grains with diffuse internal cracking, clusters of small holes, and vesicular rims previously

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420 interpreted as resulting from degassing during decarbonation of comminuted dolomite grains  
421 (Fig. 9c; Mitchell et al., 2015; Demurtas et al., 2019b).

### 423 3.5.2. Microstructures of water-dampened experiments

424 In the bulk gouges, the region furthest from the slip zone is composed of grains that  
425 show very limited fracturing and resemble the starting materials (Fig. 10a,d; compare with Fig.  
426 1e). Towards the slip zone, grains are increasingly fractured and become rounder. As in the  
427 room-humidity experiments, most of the larger “surviving” grains are composed of dolomite  
428 (Fig. 10e), consistent with data showing that calcite undergoes more efficient grain size  
429 reduction compared to dolomite (Smith et al. 2017, Demurtas et al., 2019a). Domain  
430 boundaries (e.g. between intact bulk gouge and comminuted gouge) are often gradational  
431 (Fig. 10d), and the total thickness of the comminuted zone is observed to decrease at higher  
432 slip rates (from c. 1500  $\mu\text{m}$  thick at 30  $\mu\text{ms}^{-1}$  to c. 150  $\mu\text{m}$  thick at 1  $\text{ms}^{-1}$ ). The principal slip  
433 zone consists of an ultrafine-grained matrix (grain size  $<1 \mu\text{m}$ ) composed of a mixture of calcite  
434 and dolomite, with a few well-rounded surviving dolomite grains up to 20-30  $\mu\text{m}$  in size (Fig.  
435 10b-c). At the lowest slip rate (i.e., 30  $\mu\text{ms}^{-1}$ ), the principal slip zone has a sharp but wavy  
436 boundary (characteristic wavelength of c. 300  $\mu\text{m}$ ) with the underlying gouge (Fig. 10d), and  
437 contains irregular flame-like structures defined by subtle variations in the content of calcite and  
438 dolomite (Fig. 10b). The principal slip zone is cut by discrete slip surfaces oriented subparallel  
439 to the boundaries of the gouge (Y-shear, Fig. 10c). Experiments performed at 30  $\mu\text{ms}^{-1}$  with  
440 increasing displacements (s1327, s1329, s1328, s1330-s1214 with displacements of 0.05-0.1-  
441 0.2-0.4 m, respectively; Table 1) show that the three distinct microstructural domains are  
442 already recognizable after  $<0.05$  m of slip (Fig. 10d), and that the final thickness of each  
443 microstructural domain is a function of total slip and slip rate. At a slip rate of 0.1  $\text{ms}^{-1}$ , the  
444 comminuted principal slip zone shows distinct grain size grading, characterized by an  
445 abundance of relatively large and angular dolomite particles towards the stationary side of the  
446 slip zone, and an absence of such particles towards the rotary side (Fig. 10e). Measurements  
447 of the thickness of the principal slip zone at different slip rates (Fig. 8) show a log-linear  
448 decrease in thickness from c. 400  $\mu\text{m}$  at 30  $\mu\text{ms}^{-1}$  to c. 30  $\mu\text{m}$  at 1  $\text{ms}^{-1}$ .

449 At a slip rate of 1  $\text{ms}^{-1}$ , the gouge contains an intensely comminuted c. 300-400  $\mu\text{m}$   
450 thick layer bordering the principal slip zone (Fig. 10f). The transition between the two domains  
451 is sharp and characterized in places by the occurrence of discrete Y-shears. The principal slip  
452 zone consists of lens-shaped patches of a calcite-rich and fine-grained (grain size  $<1 \mu\text{m}$ ) layer  
453 c. 30  $\mu\text{m}$  thick with negligible porosity, which is embedded in a highly comminuted and fine-  
454 grained matrix containing a few larger dolomite grains. The principal slip surface cuts sharply

Deleted: 10c; Mitchell et al., 2015; Demurtas et al., 2019b). At 1  $\text{ms}^{-1}$  and 26 MPa, the foliation was found only within 400  $\mu\text{m}$  of the principal slip surface (Supplementary Material). The principal slip zone was composed of a calcite-rich recrystallized layer, with substantially reduced porosity and well-rounded dolomite clasts a few micrometres in size (Supplementary Material)

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478 through this layer and truncates larger clasts (Fig. 10f-g). Locally, reworked angular fragments  
479 of the principal slip zone are found (Fig. 10h).

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## 481 4. Discussion

### 482 4.1. Microstructural evolution and weakening mechanisms in calcite-dolomite mixtures

483 The mechanical behaviour and microstructural evolution of calcite-dolomite gouges  
484 show substantial differences based on the availability of water during deformation (Fig. 11). In  
485 room-humidity conditions, slip strengthening at slip rates  $\leq 0.01 \text{ ms}^{-1}$  is associated with (i) initial  
486 compaction followed by dilation (Fig. 4a) and (ii) the development of a  $>500 \mu\text{m}$  thick slip zone  
487 composed of a fine-grained (c.  $1 \mu\text{m}$ ) calcite-dolomite mixture cut by Y-, R-, and  $R_1$ -shear  
488 bands (Fig. 7). These observations have previously been interpreted to relate to the  
489 development and broadening of a distributed zone of deformation during strain hardening (e.g.,  
490 Marone et al., 1990; Beeler et al., 1996; Rathbun and Marone, 2010). This is also supported  
491 by widening of the main peaks for calcite and dolomite in XRPD analysis (Fig. 6a), which is  
492 interpreted to result from a decrease in the mean crystallite size or deformation-induced  
493 microstrain within the crystallites (e.g., Ungár, 2004). A much shorter initial period of dilatancy  
494 is observed in experiments performed at  $V \geq 0.1 \text{ ms}^{-1}$  (Fig. 4a), and this correlates with (i) a  
495 transition from slip hardening to slip weakening (Figs. 2-3) and (ii) the development of a well-  
496 defined, localized principal slip zone that accommodates most of the strain after it forms (Figs.  
497 7e-f, 9 and 11) (see also Han et al., 2007a; Fondriest et al., 2013; Smith et al., 2013, 2015;  
498 Green et al., 2015; De Paola et al., 2015; Mitchell et al., 2015; Rempe et al., 2017; Pozzi et  
499 al., 2019; Demurtas et al., 2019b). The switch to slip weakening and a higher degree of strain  
500 localization is also associated with a significant temperature rise generated within the principal  
501 slip zone (Fig. 5a),  $\text{CO}_2$  emissions (Fig. 5b), and the formation of Mg-calcite and periclase in  
502 samples collected from the principal slip zone (Fig. 6c). Collectively, these observations  
503 suggest that the temperature rise at relatively high slip velocities triggered dynamic weakening  
504 and decarbonation of dolomite (and possibly calcite).

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505 At high slip rates ( $V \geq 0.1 \text{ ms}^{-1}$ ), the onset of dynamic weakening in carbonate gouges  
506 deformed at room-humidity has been interpreted as a consequence of local heating along  
507 incipient slip surfaces, which eventually coalesce into a localized and through-going shear  
508 band (De Paola et al., 2015; Smith et al., 2015; Rempe et al., 2017). Further slip then increases  
509 the bulk temperature due to continued frictional heating in the principal slip zone and  
510 dissipation of heat in to the bulk gouge, resulting in local gouge recrystallization (Smith et al.,  
511 2015). Under these conditions, high strain rates can be accommodated by temperature- and  
512 grain size-dependent deformation mechanisms leading to “viscous” flow (Green et al., 2015;

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526 De Paola et al., 2015; Pozzi et al., 2018, 2019; Demurtas et al., 2019b). Demurtas et al.  
527 (2019b) performed Transmission Kikuchi Diffraction (TKD) analysis on electron-transparent  
528 samples of the low porosity, fine-grained principal slip zone of experiment *s1221* ( $V = 1 \text{ ms}^{-1}$   
529 under room-humidity conditions; Fig. 9c) to investigate the deformation mechanisms active  
530 during coseismic sliding in calcite-dolomite mixtures. Their results show that the principal slip  
531 zone is composed of a nanogranular aggregate made of two grain populations: (i) nanograins  
532 100-300 nm in size exhibiting low internal lattice distortion, compatible with deformation by  
533 grain size sensitive creep, and (ii) nanograins >800 nm in size showing development of  
534 subgrains, suggesting deformation by grain size insensitive creep (Demurtas et al., 2019b).  
535 Although the maximum temperature measured during deformation in the present experiments  
536 was 621 °C (Fig. 5a), accommodation of the calculated strain rates ( $\dot{\gamma} = 6 \times 10^3 \text{ s}^{-1}$ ) could be  
537 explained by a significant decrease of the activation energy for creep mechanisms (and also  
538 decarbonation reactions) due to the nanogranular nature of the particles (Demurtas et al.,  
539 2019b). Similar observations were documented by Pozzi et al. (2019) in experimental  
540 nanogranular principal slip zones in pure calcite gouges deformed at coseismic slip rates.  
541 Alternatively, the temperatures achieved in the slip zones of high velocity ( $V = 1 \text{ ms}^{-1}$ ) but short  
542 duration experiments (<0.5 s) could be higher than those measured with thermocouples and  
543 estimated using numerical models. The thermocouples used here were located a few mm from  
544 the edge of the slipping zone (Fig. 1b). Additionally, they have large thermal inertia and low  
545 real acquisition rates because the electric potential developing in response to temperature  
546 changes is very slow (0.1-0.5 s) compared to the duration of the experiments (c. 0.5 s) (Sarnes  
547 and Schrüfer, 2007). Recent studies in which the temperature during experimental seismic slip  
548 was measured with optical fibres located inside the slip zone (in-situ measurements at  
549 acquisition rates of 1 kHz) detected temperatures 300-400 °C higher than those measured with  
550 thermocouples (Aretusini et al., 2019). Temperatures in the slipping zone substantially higher  
551 than 621 °C would render grain size- and temperature-dependent deformation mechanisms  
552 more efficient. Instead, in the case of experiment *s1218* performed at  $V = 0.1 \text{ ms}^{-1}$ , the  
553 moderate dynamic weakening ( $\mu_{ss} = 0.55$ ) can be related to more limited frictional heating  
554 within the principal slip zone both in time (max temperature measured of 190 °C, Fig. 5a) and  
555 space (patchy recrystallized areas in Fig. 7e-f). However, at least locally, the temperature  
556 increase was sufficiently large to decompose dolomite (i.e. c. 550 °C), as testified by the clear  
557  $\text{CO}_2$  peak measured during shearing at this velocity (Fig. 5b).

558 In water-dampened conditions, the mechanical behaviour of the calcite-dolomite  
559 mixtures is similar (slight slip strengthening to slip neutral) at all slip rates up to  $0.1 \text{ ms}^{-1}$  (Fig.  
560 2c). The thickness of the principal slip zone decreases log-linearly with increasing slip rate,

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569 indicating a progressively higher degree of localization (Fig. 8 and 11). However, this has no  
570 obvious effect on the steady state friction coefficient (Fig. 3b), possibly suggesting that the  
571 steady state is controlled by strain and that strain is kept constant by microstructural  
572 reorganization distributed within the slip zone. The principal slip zone is composed of a very  
573 fine-grained ( $\ll 1 \mu\text{m}$ ) matrix of calcite and dolomite that includes a few well-rounded dolomite  
574 clasts up to 20-30  $\mu\text{m}$  in size (Fig. 10). The similarity in microstructure at all investigated slip  
575 rates suggests that water has a major role in promoting faster grain size reduction at the onset  
576 of slip, possibly by decreasing the surface energy and yield stress of calcite and dolomite  
577 (Risnes et al., 2005; Røyne et al., 2011). XRPD analysis of the slip surface of experiment  
578 *s1214* ( $30 \mu\text{ms}^{-1}$ ) showed the formation of aragonite (Fig. 6b). Given that the starting materials  
579 were composed of calcite and dolomite only, the aragonite must have formed during  
580 deformation. Li et al. (2014) documented polymorphic transformation of calcite into aragonite  
581 due to mechanical grinding in a dry (i.e., room humidity) environment. Our observations  
582 therefore suggest that relatively dry patches could develop in the gouge layer during slip (or  
583 were present at the onset of slip), or that such transformation is also possible under water-  
584 dampened conditions.

585 In water-dampened experiments at  $1 \text{ ms}^{-1}$ , abrupt dynamic weakening preceded by a  
586 very short-lived strengthening phase has previously been documented in experiments on  
587 calcite gouges (Rempe et al., 2017, 2020) and calcite marbles (Violay et al., 2014). In gouges,  
588 Rempe et al. (2017, 2020) suggested that the rapid onset of dynamic weakening could be  
589 related to faster grain size reduction in the presence of water, leading to an early switch from  
590 brittle deformation to grain size sensitive creep in the principal slip zone, analogous to the  
591 processes suggested to occur in dry gouges (De Paola et al., 2015; Demurtas et al., 2019b;  
592 Pozzi et al., 2019). However, there is an apparent discrepancy between the relatively low  
593 maximum temperature measured close to the principal slip zone in water-dampened  
594 experiments ( $200 \text{ }^\circ\text{C}$  at  $V = 1 \text{ ms}^{-1}$ ; Fig. 5a), and the observed  $\text{CO}_2$  production (Fig. 5b)  
595 combined with microstructural evidence for recrystallization during deformation (Fig. 10f-g). As  
596 previously discussed, this could be due to an underestimate of the peak temperature (Aretusini  
597 et al., 2019). Alternatively, vaporization of water during coseismic sliding could buffer the  
598 temperature due to the endothermic nature of the phase transition (Chen et al., 2017a, b). If  
599 the pore pressure increase is sufficiently large, then fluids (and vapour) could pressurize the  
600 gouge layers and play an important role during dynamic weakening. Finally, Ohl et al. (2020)  
601 proposed that mechanical liming (see Martinelli and Plescia, 2004) along natural faults could  
602 be a possible slip weakening mechanism that does not necessarily involve a macroscopic  
603 temperature increase of  $>500\text{-}600 \text{ }^\circ\text{C}$ .

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609 **4.2. Implications for natural fault zones**

610 **4.2.1 Gouge fluidization at slip rates between 30  $\mu\text{ms}^{-1}$  and 0.1  $\text{ms}^{-1}$**

611 The slip zone of the water-dampened experiment performed at 30  $\mu\text{ms}^{-1}$  is  
 612 characterized by flame-like structures, and domain boundaries that display a characteristic  
 613 wavelength (Figs. 10b,d, 11). Similar structures are typical of soft sediment deformation (Allen,  
 614 1985) and have also been described within fault cores (Brodsky et al., 2009), where they are  
 615 interpreted to result from grain mobilization promoted by fluid overpressure and a difference in  
 616 viscosity between two adjacent layers during deformation. Additionally, the occurrence of grain  
 617 size grading within the water-dampened principal slip zone formed at 0.1  $\text{ms}^{-1}$  is indicative of  
 618 grain rearrangement due to frictional sliding (see Masoch et al., 2019 and reference therein),  
 619 referred to as the “Brazil nut” effect, a phenomenon observed when large grains move to the  
 620 top of a fluidized layer due to differences in dispersal pressure between large and small  
 621 particles (Williams, 1976). Grain size grading was reported by Boullier et al. (2009) from the  
 622 principal slip zone of the 1999  $M_w$  7.6 Chi-Chi earthquake in Taiwan, and from exhumed normal  
 623 faults in Alpine Corsica (Masoch et al., 2019). Additionally, similar microstructures were  
 624 produced by Boulton et al. (2017) in high velocity rotary-shear experiments ( $V = 1 \text{ ms}^{-1}$ )  
 625 performed on clay-rich drill chips retrieved from the Alpine Fault (New Zealand). As in the  
 626 experiments by Boulton et al. (2017), grain size grading in our experiments was observed only  
 627 in water-dampened conditions. Collectively, the presence of flame-like structures, undulating  
 628 domain boundaries with a characteristic wavelength, and grain size grading, suggests that  
 629 water-dampened gouges experienced fluidization at slip rates between 30  $\mu\text{ms}^{-1}$  and 0.1  $\text{ms}^{-1}$ .  
 630 This is significant because textures and microstructures related to fluidization in natural  
 631 gouges and cataclases are often interpreted to form during coseismic slip at high velocities  
 632 (e.g., Monzawa and Otsuki, 2003; Rowe et al., 2005; Boullier et al., 2009; Brodsky et al., 2009;  
 633 Demurtas et al., 2016; Boulton et al., 2017; Smeraglia et al., 2017). However, our observations  
 634 suggest that fluidization might occur at lower slip velocities if local fluid pressures can build up  
 635 in deforming gouge layers, and may not necessarily be an indicator of coseismic slip.

636 Various mechanisms have been proposed to account for fluidization of granular  
 637 materials in fault zones, including (i) frictional heating and thermal pressurization (Boullier et  
 638 al., 2009), (ii) dilation that limits grain-grain contacts (Borradaile, 1981; Monzawa and Otsuki,  
 639 2003), and (iii) focussed fluid flow along slip zones during and after coseismic sliding (e.g.,  
 640 fault-valve mechanism of Sibson, 1990). In our experiments, temperature measurements  
 641 made during slip at 30  $\mu\text{ms}^{-1}$  suggest that significant frictional heating is unlikely, and therefore  
 642 thermal pressurization is an unlikely mechanism within the slip zone. Water-dampened

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650 experiments are characterized by continuous compaction, which also excludes the dilation-  
 651 related hypothesis of Borradaile (1981). ~~The rapid grain size reduction occurring at the onset~~  
 652 ~~of slip, in particular for calcite, creates a~~ principal slip zone ~~that could readily accommodate~~  
 653 continuous compaction. ~~Within the principal slip zone, local variations in grain size could create~~  
 654 ~~transient differences in gouge permeability that may promote pore fluid pressure build up.~~ A  
 655 sudden release of ~~fluid~~ from pressurized ~~patches~~ could result in gouge mobilization and  
 656 injection of material ~~into~~ the adjacent ~~regions~~. ~~Such variations in permeability and pore~~  
 657 ~~pressure are likely to occur on a local level that may not be recorded by bulk variations of~~  
 658 ~~gouge thickness during deformation.~~

#### 659 **4.2.2 Foliation development in calcite-dolomite gouges at coseismic slip rates**

661 Foliated gouges and cataclasites are common fault rocks in the brittle upper crust  
 662 (Snoko et al., 1998). Typically, they are interpreted to form due to a combination of cataclasis  
 663 and dissolution-precipitation reactions during aseismic fault creep (e.g., Rutter et al., 1986;  
 664 Chester and Chester, 1998; Lin, 2001; Collettini and Holdsworth, 2004; Jefferies et al., 2006;  
 665 De Paola et al., 2008; Wallis et al., 2013). Experimental observations support this idea, and  
 666 show that well-defined foliations can form as a result of dissolution-precipitation reactions  
 667 accompanied by granular flow and frictional sliding at low slip rates ( $V < 1 \mu\text{ms}^{-1}$ ; Bos et al.,  
 668 2000; Niemeijer and Spiers, 2006).

669 However, the association of foliated fault rocks with possible microstructural indicators  
 670 of seismic slip in natural fault rocks (e.g., ~~slip zones containing recrystallized material~~, see  
 671 Demurtas et al., 2016) led Smith et al. (2017) to investigate the possibility that some foliated  
 672 gouges and cataclasites might have a coseismic origin. Rotary-shear experiments performed  
 673 at a slip rate of  $1.13 \text{ ms}^{-1}$  on gouges composed of 50 wt.% calcite and 50 wt.% dolomite showed  
 674 the development of a foliation defined by an organized banding of heavily fractured calcite and  
 675 dolomite clasts (Smith et al., 2017). Experiments performed at increasing displacements  
 676 revealed that the foliations are established during the initial strengthening phase, when  
 677 distributed strain throughout the bulk gouge causes grain comminution and distributed  
 678 shearing. Once dynamic weakening occurs, strain progressively localizes into a single  
 679 continuous principal slip zone, and the foliation in the bulk gouge does not show any further  
 680 microstructural change. Shear strain analysis of the foliated layers showed that relatively low  
 681 values of strain ( $\gamma < 4$ ) are needed to develop a foliation. Based on their observations, Smith  
 682 et al. (2017) suggested that some natural foliated gouges and cataclasites characterized by  
 683 compositional banding, grain size variations, and preferred particle or fracture alignments,  
 684 could form by distributed brittle flow as strain localizes during coseismic shearing, especially if

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**Moved up [2]:** and calcite marbles (Violay et al., 2014). In

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**Moved up [4]:** suggested to occur in dry gouges (De Paola et al., 2015; Demurtas et al., 2019b; Pozzi et al., 2019).

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**Moved up [6]:** peak temperature (Aretusini et al., 2019).

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**Moved up [7]:** ( ) proposed that mechanical liming (see Martinelli and Plescia, 2004) along natural faults could be a possible slip weakening mechanism that does not necessarily involve a macroscopic temperature increase of  $>500\text{-}600 \text{ }^\circ\text{C}$ .

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727 such foliations are found in proximity to microstructural indicators of coseismic slip. The  
728 experiments presented in this paper allow us to test this hypothesis over a wider range of slip  
729 rates (i.e.,  $30 \mu\text{ms}^{-1} - 1 \text{ms}^{-1}$ ) and deformation conditions (i.e., room humidity vs. water  
730 dampened). [In these new experiments, the](#) formation of well-defined foliations throughout the  
731 bulk gouge was only observed at high slip rates ( $V = 1 \text{ms}^{-1}$ ) and room humidity conditions  
732 ([Figs. 9, 11](#)), corresponding to the conditions presented in Smith et al. (2017). Local foliation  
733 development was also observed in lower velocity experiments at room humidity, although this  
734 was restricted to regions  $<400 \mu\text{m}$  from the principal slip surface (Fig. [7e](#)).

735 Our new experiments support the hypothesis presented in Smith et al. (2017) that well-  
736 defined foliations in calcite-dolomite gouges can form during high velocity sliding in carbonate  
737 gouges, and could be used in conjunction with other microstructures (e.g., seismic slip  
738 indicators) to better understand localization processes in faults. The lowest slip velocity studied  
739 here (i.e.,  $30 \mu\text{ms}^{-1}$ ) is still too high for pressure-solution to be efficient in calcite or dolomite [at](#)  
740 [room temperature](#). Lower slip rates might promote the activation of pressure-solution, which  
741 could result in the formation of a foliation under certain conditions. Grain elongation and  
742 foliation development have previously been reported in experiments performed on calcite-  
743 dolomite mixtures by Delle Piane et al. (2009) [and on calcite by Verberne et al. \(2017\)](#).  
744 However, in [the former](#) case, the deformation conditions (i.e., torsion experiments at  
745 temperatures of 700-800 °C, confining pressure of 300 MPa, shear strain rate of  $3 \times 10^{-4}$ - $10^{-5}$   
746  $\text{s}^{-1}$ ) were representative of mid- to lower crustal depths, rather than the low-pressure low-  
747 temperature ambient conditions explored here. [Although the slip rates in Verberne et al. \(2017\)](#)  
748 [were closer to the ones explored in this study \(i.e.,  \$1 \text{ms}^{-1}\$  to  \$100 \mu\text{ms}^{-1}\$ \), the temperature \( \$T\$   
749  \$= 550 \text{ °C}\$ \) and stress conditions \(effective normal stress of 50 MPa and pore fluid pressure of  
750 100 MPa\) were designed to investigate the ductile-brittle transition at the base of the  
751 seismogenic zone rather than shallow faulting.](#)

## 753 5. Conclusions

754 A series of rotary-shear experiments was performed on gouges composed of 50 wt.%  
755 calcite and 50 wt.% dolomite to develop an understanding of microstructural evolution at a  
756 range of slip rates ( $30 \mu\text{ms}^{-1} - 1 \text{ms}^{-1}$ ), fluid conditions (room humidity and water dampened),  
757 total displacements (0.05–0.4 m), and normal [load of 17.5 MPa](#).

758 The evolution of the apparent friction coefficient is strongly influenced by the presence  
759 of water. [Under room humidity conditions](#), slip strengthening is observed up to slip rates of  
760  $0.01 \text{ms}^{-1}$ , above which dynamic weakening occurs. In water-dampened conditions, slight slip  
761 strengthening to slip neutral friction characterises experiments up to slip velocities of  $0.1 \text{ms}^{-1}$ ,

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778 above which dynamic weakening occurs abruptly. The mechanical differences observed under  
779 room-humidity and water-dampened conditions are also reflected in the microstructures of the  
780 deformed gouge layers. At room humidity, slip strengthening is associated with diffuse  
781 deformation and the development of a relatively thick slip zone cut by Y-, R-, and R<sub>1</sub>-shear  
782 bands. The onset of dynamic weakening is concomitant with the development of a localised  
783 principal slip zone containing evidence of dolomite decarbonation and calcite recrystallization.  
784 In the presence of water, evidence of gouge fluidization within a fine-grained principal slip zone  
785 is observed at slip rates from 30 μms<sup>-1</sup> to 0.1 ms<sup>-1</sup>, suggesting that fluidization may not be  
786 restricted to coseismic slip rates. At 1 ms<sup>-1</sup>, the principal slip zone is characterised by patches  
787 of recrystallized calcite that are locally broken and reworked.

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788 The development of a well-defined foliation in the bulk gouge layer only occurs in room-  
789 humidity experiments at a slip rate of 1 ms<sup>-1</sup>, consistent with the work of Smith et al. (2017).  
790 This observation supports the notion that some foliated gouges and cataclasites may form  
791 during coseismic slip in natural carbonate-bearing faults.

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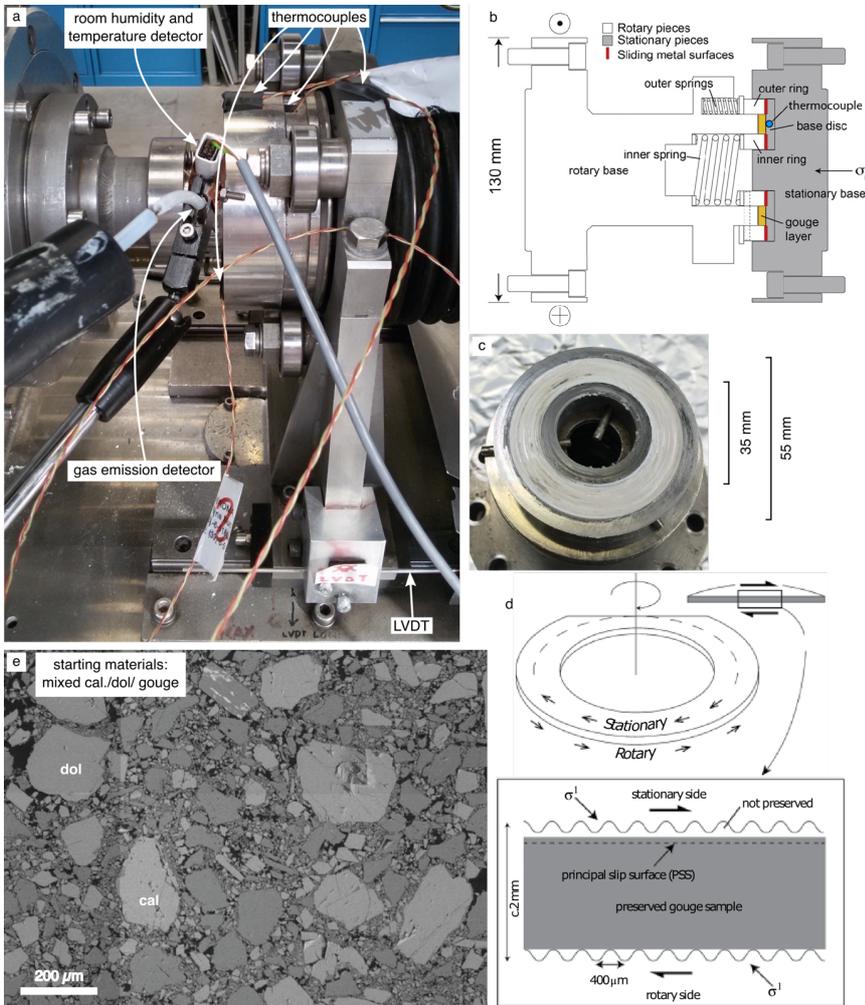
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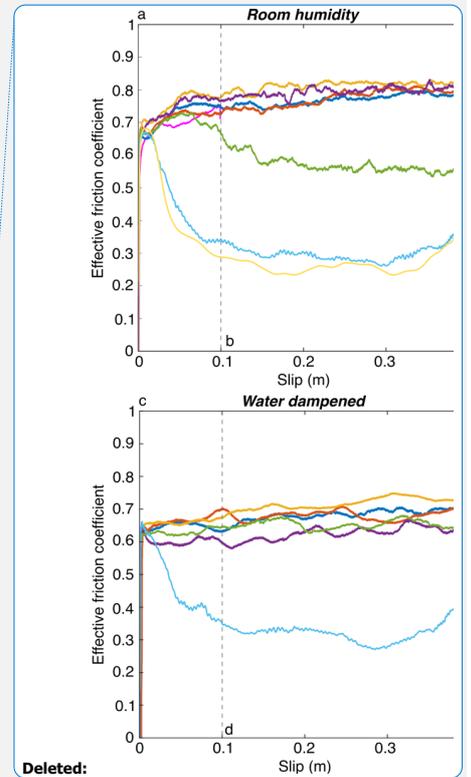
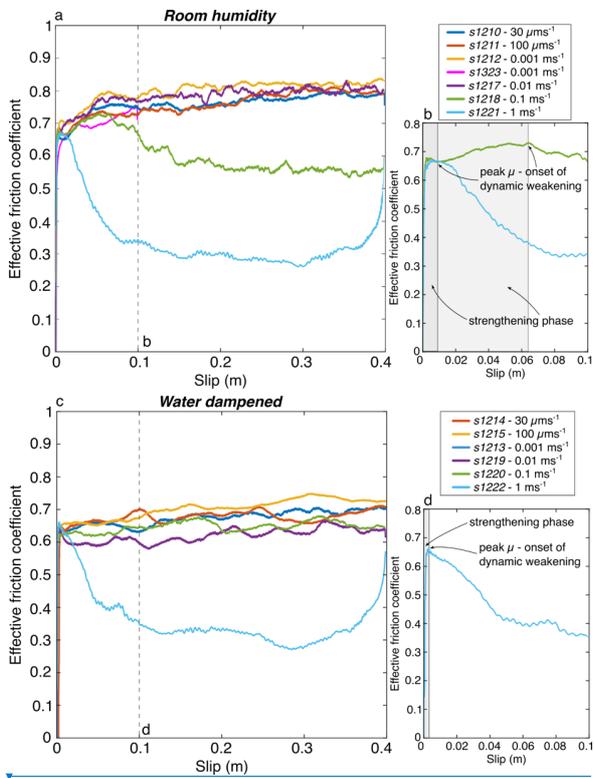
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 1227 **Figure 1. Rotary-shear experimental setup.** a) Detectors in the sample chamber. Gas  
 1228 emission, humidity, and temperature detectors were placed at < 1 cm from the sample holder.  
 1229 Four thermocouples were placed on the stationary side at increasing distances from the gouge  
 1230 layer. Note: the position of the thermocouple nearest to the gouge layer is not visible here and  
 1231 is illustrated in b). b) Diagram of the gouge holder with the location of the thermocouple nearest  
 1232 to the gouge layer (modified after Smith et al., 2015). c) Sample appearance post deformation  
 1233 with mirror-like slip surface formed in an experiment performed at  $V = 0.1 \text{ ms}^{-1}$ . d) Diagram  
 1234 showing the location of the recovered and analysed gouge layer after the experiment (after  
 1235 Smith et al., 2017). e) SEM backscattered electron (SEM-BSE) image of the starting material  
 1236 after applying 17.5 MPa for 300 s.  
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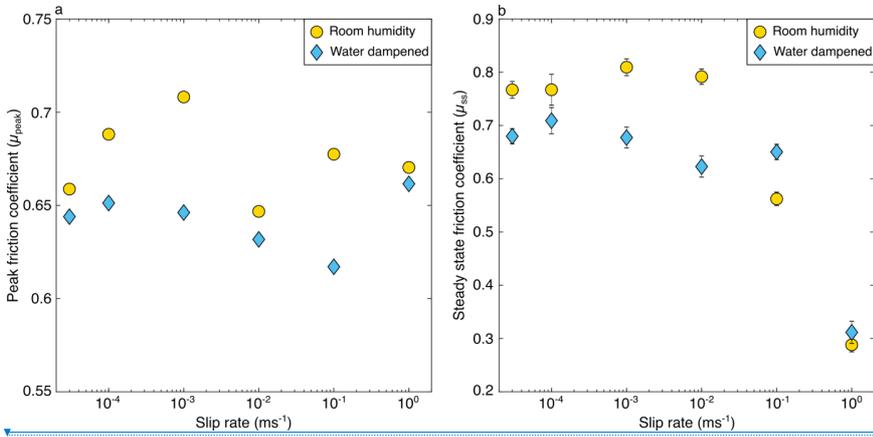
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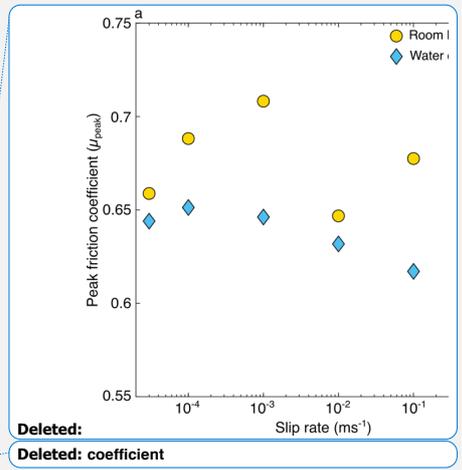
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**Figure 2. Effective friction coefficient in mixed calcite-dolomite gouges.** a) and c) Effective friction coefficient versus slip under room humidity and water dampened conditions. c) and d) Detail of effective friction coefficient versus slip in the first 0.1 m of slip in experiments where slip-weakening was observed.

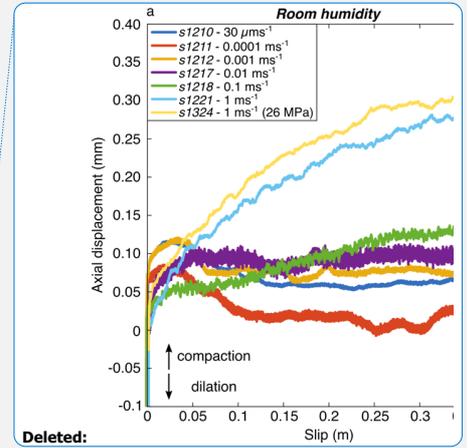
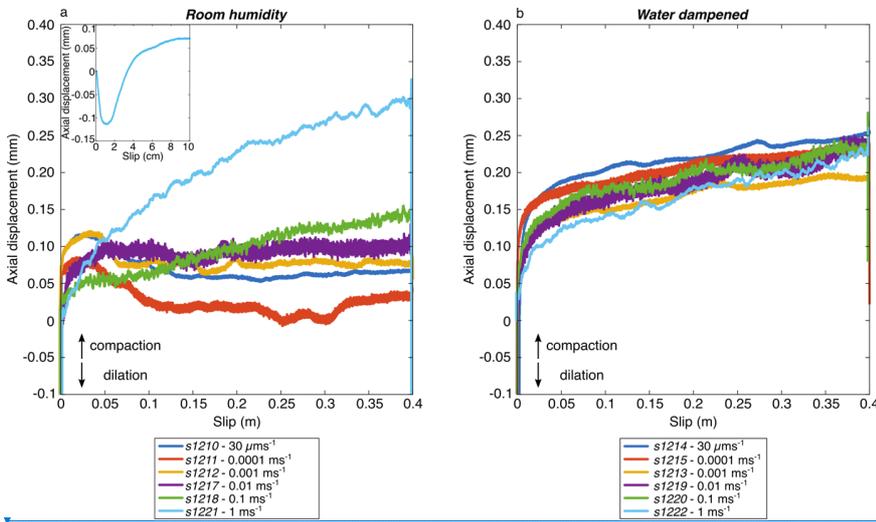


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**Figure 3. Peak and steady state friction coefficients.** a) For experiments that showed slip strengthening, the peak friction coefficient was calculated just before the onset of strengthening behaviour in the first 0.1 m of slip. For experiments showing dynamic weakening, the peak friction immediately precedes the friction drop (see Fig. 2b and 2d). b) Steady state friction coefficient was calculated at displacements between 0.15 m and 0.35 m.



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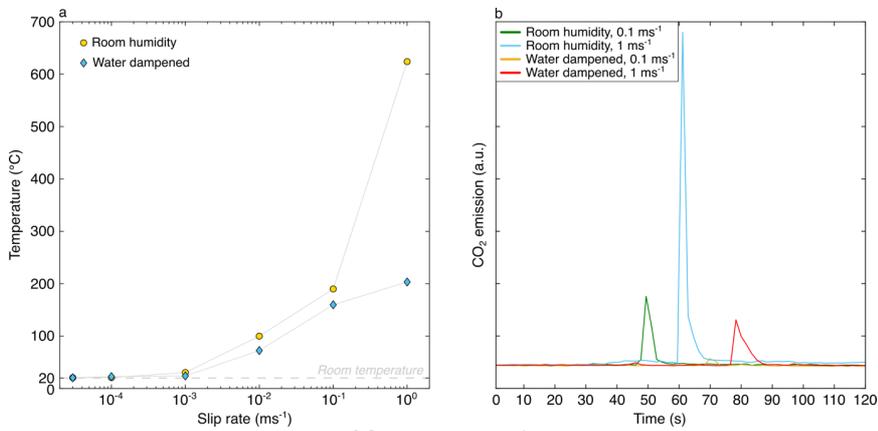
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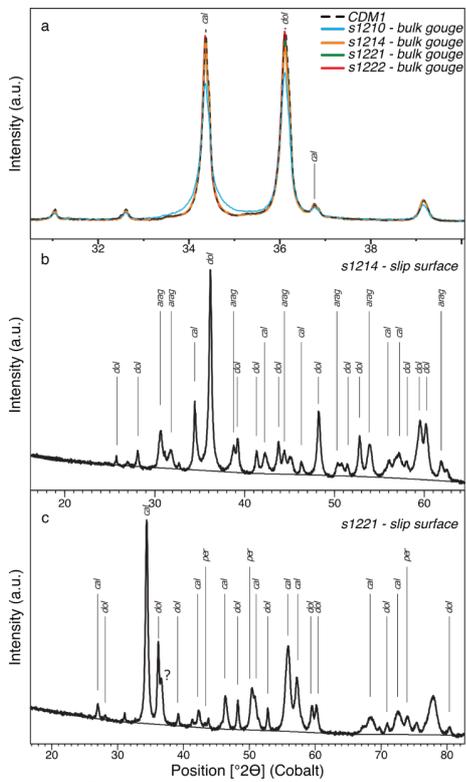
**Figure 4. Gauge thickness evolution with slip and slip rate.** a) Under room humidity conditions and for slip rates of  $V \leq 0.01 \text{ ms}^{-1}$ , an initial compaction phase was followed by dilation (lasting 0.1-0.15 m) and then constant thickness. At higher slip rates ( $V \geq 0.1 \text{ ms}^{-1}$ ), after a short initial dilatancy phase, the gouge compacted constantly throughout the whole experiment, with compaction rate increasing with slip rate. b) Under water dampened conditions, the gouge compacted at a similar rate at all investigated slip rates.



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**Figure 5. Peak temperatures and  $\text{CO}_2$  emissions.** a) Maximum temperature measured by the thermocouple located closest to the gouge layer (see Fig. 1b for location). b)  $\text{CO}_2$  emissions for experiments at  $V \geq 0.1 \text{ ms}^{-1}$  in both room-humidity and water-dampened conditions. Greater emissions occur at room-humidity conditions, but smaller and distinct peaks are also observed in the presence of water.

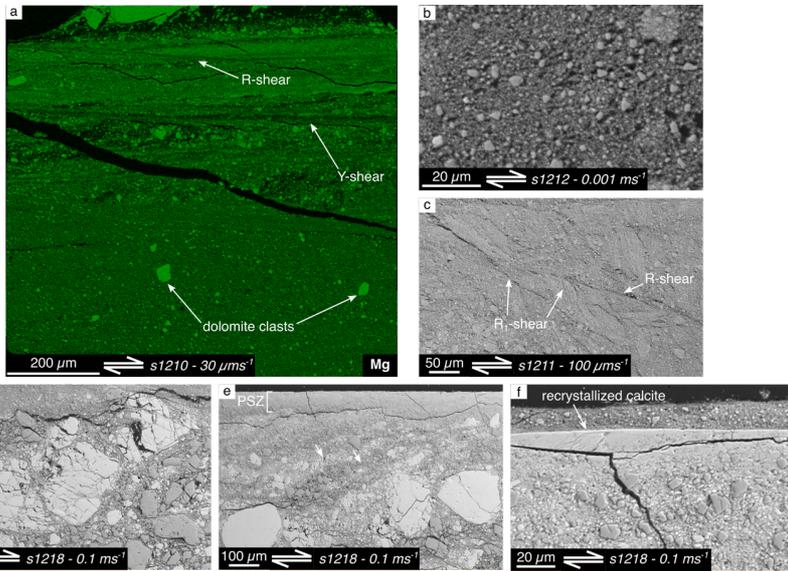
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1281  
 1282 **Figure 6. XRPD analysis of bulk gouge and slip surfaces.** a) Bulk gouge shows a  
 1283 Lorentzian profile for the main calcite peak in experiment *s1210*, suggesting either a large  
 1284 crystallite size distribution or microstrain as a result of intense comminution involving a large  
 1285 fraction of the gouge. b) At  $30 \mu\text{ms}^{-1}$  in water-dampened conditions, traces of aragonite are  
 1286 found on the slip surface as a result of calcite polymorphic transformation during prolonged  
 1287 mechanical grinding. c) For *s1221* ( $1 \text{ms}^{-1}$  in room-humidity conditions), presence of Mg-calcite  
 1288 and periclase (MgO) on the mirror-like slip surface is observed due to decarbonation of  
 1289 dolomite.  
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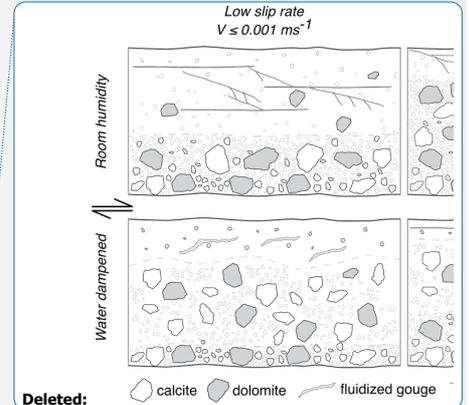
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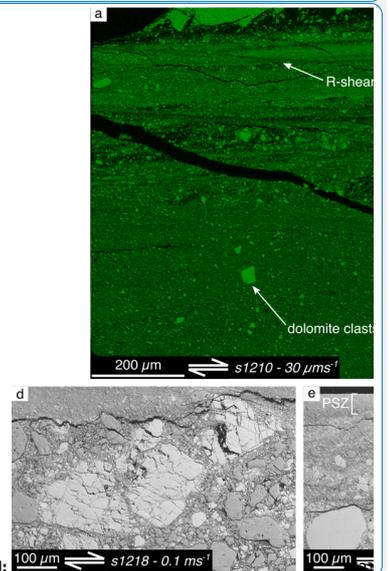
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**Figure 7. Microstructures of experiments in room-humidity conditions and  $V \leq 0.1 \text{ ms}^{-1}$ .**  
 a) Mg element map of the thick slip zone highlighting the location of remnant dolomite clasts.  
 b) Detail of the fine-grained matrix in the slip zone made of a calcite-dolomite mixture with surviving sub-rounded dolomite clasts up to few tens of micrometres in size. c) The fine-grained slip zone is commonly cut by Y-, R- and  $R_1$ -shear bands crosscutting each other. d) Enhanced grain size reduction in calcite grains due to fracturing along cleavage planes. e) Development of a weak foliation adjacent to the principal slip zone at  $V = 0.1 \text{ ms}^{-1}$ . f) Patches of dynamically recrystallized calcite along the principal slip zone at  $V = 0.1 \text{ ms}^{-1}$ .

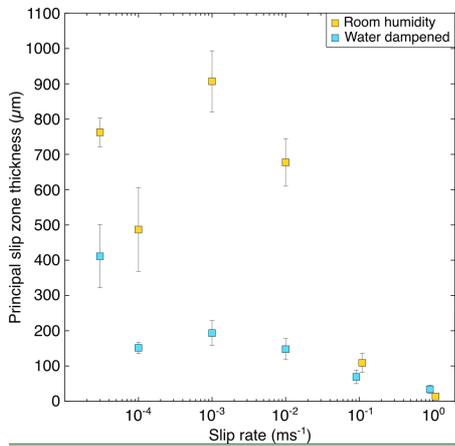


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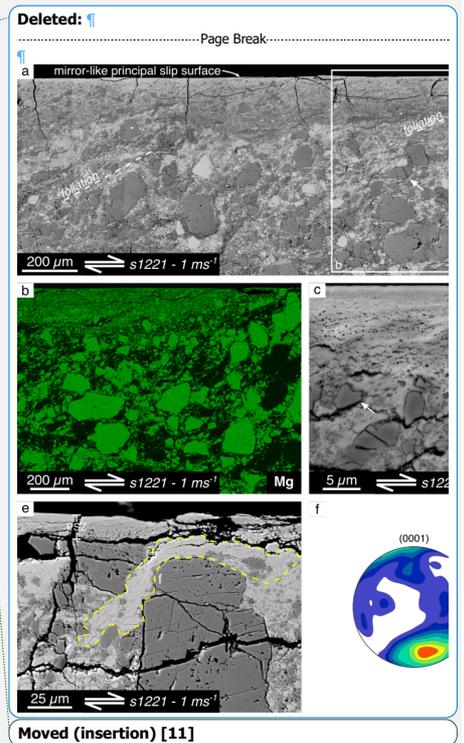


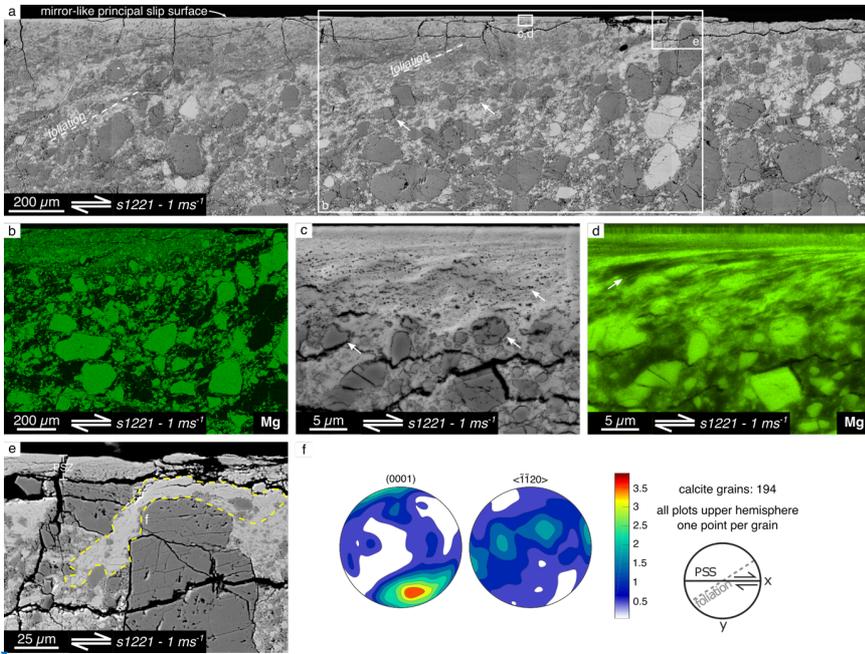
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**Figure 8. Slip zone thickness evolution with slip rate and presence of water. The thickness of the localized slip zone decreases almost linearly with  $\log(V)$  in water-dampened experiments. For room-humidity conditions, partial sample loss after the experiment means slip zone thickness values are a minimum estimate.**

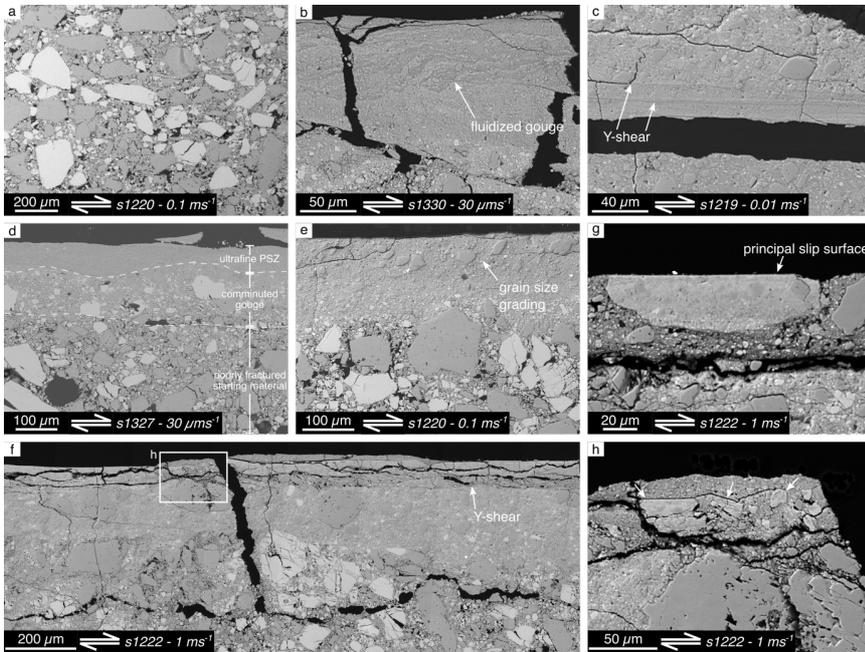




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 1336 **Figure 9. Microstructures of experiments in room-humidity conditions and  $V = 1 \text{ ms}^{-1}$ .** a)  
 1337 Development of a foliation consisting in alternation of calcite- and dolomite-rich domains,  
 1338 antithetically inclined c.  $40^\circ$  from the PSS and becoming subparallel to the gouge boundaries  
 1339 when approaching the PSS. Larger dolomite (and when present calcite) clasts have tails of  
 1340 fine-grained material, resembling mantled porphyroclasts. b) Mg element map highlighting  
 1341 foliation development in the bulk gouge. c) Dolomite clasts adjacent to the principal slip zone  
 1342 are characterized by internal holes and vesicular rims interpreted as due to degassing during  
 1343 dolomite decarbonation reaction. Banding of low and higher porosity in the principal slip zone  
 1344 is an indicator for dolomite content. d) Mg element map of c) showing dolomite and calcite  
 1345 banding in the principal slip zone. e) Transition from the principal slip zone to the underlying  
 1346 fractured and foliated gouge with calcite showing evidence of dynamic recrystallization. f)  
 1347 Orientation data for calcite in area highlighted in e). showing the development of a clear CPO  
 1348 along the c-axes.  
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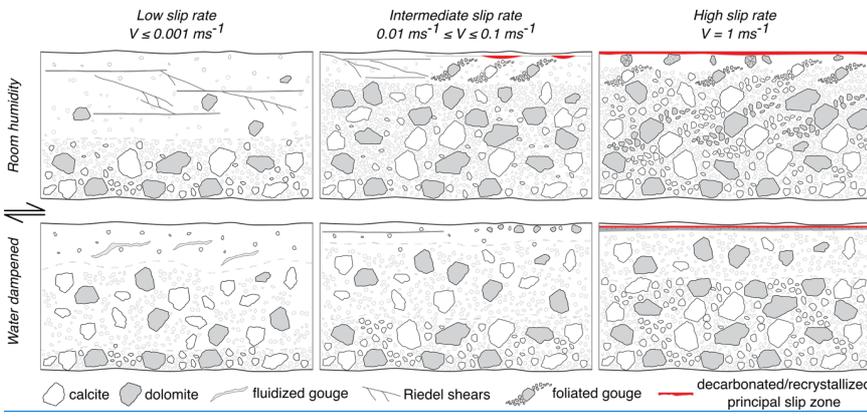
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**Figure 10. Microstructures of experiments in water-dampened conditions.** a) The bulk gouge is made of poorly fractured calcite and dolomite mixture highly resembling the starting material. b) Occurrence of fluidized structures in the principal slip zone of experiments performed at  $30 \mu\text{ms}^{-1}$ . c) At  $0.0001 \text{ ms}^{-1} \leq V < 0.1 \text{ ms}^{-1}$ , the principal slip zone is cut by multiple Y-shears. d) In the presence of fluid water, an ultrafine principal slip surface develops, overlying a highly comminuted gouge which then transitions to a poorly fractured starting material. e) At  $V = 0.1 \text{ ms}^{-1}$ , grain size grading is observed in the principal slip zone, with larger clasts occurring near the principal slip surface. f) At  $V = 1 \text{ ms}^{-1}$ , strain localizes on a compacted, low porosity, recrystallized slip zone, which g) is not continuous and h) often found broken and reworked.

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**Figure 11. Summary of microstructural evolution at different slip rates and deformation conditions.**

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