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JGR Solid Earth

RESEARCH ARTICLE

10.1029/2025JB032629

Key Points:

- Fluids promote healing and relaxation in terms of absolute values and rates
- Initial mean grain size controls the PSZ-to-bulk-gouge thickness ratio, which in turn modulates relaxation
- Healing is independent of the initial mean grain size and may reflect the role of the final mean grain size in the PSZ

Supporting Information:

Supporting Information may be found in the online version of this article.

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Grain Size and Fluid Effects on the Frictional Behavior of Simulated Carbonate Fault Gouge

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Abstract Carbonate rocks exhibit a span of clast and grain size in fault zones. However, the effect of grain size distribution on the frictional behavior of carbonate faults and its implications for natural and induced seismicity remain poorly understood. This study elucidates the effect of grain size distribution on the frictional behavior of Lower Carboniferous (Dinantian) simulated fault gouge, relevant to geothermal energy exploitation in Northwest Europe. We tested samples in a rotary shear apparatus under air dry and drained conditions using deionized (DI) water or geothermal brine. In each experiment, we stepped the normal stress from 1 to 10 MPa and back, in 1 MPa intervals. At each interval, we performed a slide-hold-slide procedure with 10 s slide time, 10-100-1000 s hold times, and shear velocity of 20 $\frac{\mu\text{m}}{\text{s}}$. Our results demonstrated that the presence of fluids in carbonate gouges increased the creep coefficient of internal friction, healing, and relaxation (absolute values and rates), consistent with the effects of pressure solution and corroborated by the CNS microphysical modeling. Additionally, frictional sliding facilitated by water films along grain boundaries controlled the peak and steady-state coefficients of friction. Healing was independent of the initial grain size distribution and occurred in the PSZ. Relaxation occurred both in the PSZ and bulk gouge and was governed by PSZ thickness, which positively correlated with the initial mean grain size.

Plain Language Summary Geological fault zones, where earthquakes can take place, contain gouges with a range of grain sizes. However, how the relative amount of large versus small grains changes the earthquake behavior is not well understood. In this study, we investigated how the grain size of limestone fault rocks affected their earthquake behavior. We used an important rock type for the development of geothermal energy in Northwest Europe. We used a ring-shear apparatus to simulate the earthquake cycle, with dry, plain water, or geothermal saltwater samples. Our results showed that the presence of fluids mattered in terms of absolute strength and how fast a fault regained or lost strength after sliding. The grain size did change how much and how fast a fault could lose strength between simulated earthquakes. That was caused by the size of the area where the deformation happened, in a narrow zone, called the principal slip zone. However, grain size did not change how much and how quickly faults regained strength. That happened because the grain size in those narrow zones ended up being similar, no matter what the original grain sizes were.

1. Introduction

Natural fault zones comprise low-strain and high-strain zones, commonly called the damage zone and fault core, respectively (Caine et al., 1996; Childs et al., 2009; Torabi et al., 2019; Wibberley et al., 2008). The fault core accommodates most of the displacement and deformation in the fault zone domain. It may include localized shear surfaces and significant accumulations of granular wear material such as breccia, clasts and lenses of the host rock, clay smear (silicate or carbonate), fault gouge, and diagenetic features (Bastesen et al., 2013; Braathen et al., 2009; Collettini et al., 2009; Faulkner et al., 2003; Fredman et al., 2007; Giorgetti et al., 2015; Schultz & Fossen, 2008; Torabi & Berg, 2011; Torabi et al., 2019). Specifically, fault gouge is formed by progressive shearing, crushing, and frictional wear of host rocks (Bastesen & Braathen, 2010; Chester & Chester, 1998; Childs et al., 2009; Collettini et al., 2009; Hunfeld et al., 2017; R. H. Sibson, 1986). In laboratory experiments, powdered rock material is frequently used to simulate natural fault gouge (Marone & Scholz, 1989). Geological fault zones that experienced significant displacement exhibit not only certain particle size distributions (An & Sammis, 1994; Biegel et al., 1989; Steacy & Sammis, 1991; Storti et al., 2003), but also internal structures, for example, a Riedel shear zone structure (Logan, 1979). Significant displacement is needed to increase the maturity

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of such analogue fault gouges. This can be achieved using a rotary shear apparatus (Bishop et al., 1971; Bromhead, 1979).

Slide-hold-slide (SHS) tests (Dieterich, 1972) provide a rough approximation for the seismic cycle in the sense that slip accumulates during the “slide” portion of the test and then, during the hold period, the fault creeps, and frictional strength recovers, similar to postseismic sliding and interseismic fault healing. The increase in frictional strength depends on various factors such as normal stress, shear stress, loading rate, fault slip rate, temperature, fault zone mineralogy, and presence of water as summarized and reported by Noël et al. (2024), where fault zone lithology (Arts et al., 2024; B. M. Carpenter, Collettini, et al., 2016; Saffer & Marone, 2003; Seyler et al., 2023), and presence and chemical activity of fluids (B. Bos et al., 2000; Dieterich & Conrad, 1984; Niemeijer et al., 2008; Pluymakers & Niemeijer, 2015) are critical factors. Healing is usually assumed to be log-linearly dependent on the duration of the periods of zero imposed slip (interseismic periods) (Bos & Spiers, 2002a; Dieterich, 1972; Hunfeld et al., 2020; Marone, 1998; Niemeijer et al., 2008; Olsen et al., 1998; Pluymakers & Niemeijer, 2015; Yasuhara et al., 2005), but also on fault zone lithology (Arts et al., 2024; B. M. Carpenter, Collettini, et al., 2016; Saffer & Marone, 2003; Seyler et al., 2023), and presence and chemical activity of fluids (B. Bos et al., 2000; Dieterich & Conrad, 1984; Niemeijer et al., 2008; Pluymakers & Niemeijer, 2015). A similar log-time dependence of relaxation has been observed for studies in fault gouge material (Bocquet et al., 1998; B. M. Carpenter, Collettini, et al., 2016; H. Jaeger et al., 1989; Knight et al., 1995; Linz & Döhle, 1999; Peng & Ohta, 1998; Snieder et al., 2017) and post-seismic deformation (Avouac, 2015; Benioff, 1951; Griggs, 1939; Savage et al., 2005).

Fault reactivation can occur either by perturbing the effective normal stress on the fault itself or by altering the fault properties. Given that carbonates are relatively reactive rocks, the injection of chemically reactive fluids, such as CO₂, have a relatively significant impact compared to other crustal reservoir rocks (Rohmer et al., 2016). Temperature changes or changes in fluid chemistry resulting from fluid injection in the subsurface may as well perturb the friction coefficient (Kang et al., 2019). Carbonate minerals like dolomite and calcite, exhibit an increase in solubility with decreasing temperature (Langmuir, 1997). Pluymakers et al. (2016) compared the effect of temperature for anhydrite versus carbonate (calcite and dolomite) laboratory faults, which showed a similar evolution of frictional properties with temperature. Anhydrite, like carbonates, exhibits a relatively high solubility at low temperatures with an inverse temperature dependence on solubility (Pluymakers & Niemeijer, 2015). Additionally, carbonates are prone to deformation by fluid-assisted diffusive processes such as pressure solution (B. A. Verberne et al., 2013). Moreover, Seithel et al. (2019) hypothesized that in the German Molasse basin, chemical processes of carbonate rock dissolution might partially explain features like the time delay in induced seismic events compared to the timing of the injection operations. This is in line with Kang et al. (2019), who proposed that long-term chemical processes can reduce fault strength, thus increasing fault reactivation potential. Finally, in mature faults, where the grain size is smaller compared to a fault that has not experienced substantial strain, reaction rates are likely to be increased (Niemeijer et al., 2008). Should the chemical reactions occur, first, the fine-grained portion of a fault gouge would be affected, and the proportion of small versus coarse grains would change. This, in turn, would affect the rates at which processes like pressure solution would occur, which can then also affect frictional behavior.

A few studies have reported the effects of the grain size distribution of the gouge on frictional fault healing and relaxation. Casas et al. (2025) suggested that uniform grain size distributions positively correlate with increasing thickness of deformation zones, which are critical for laboratory earthquake nucleation. Additionally, they proposed that in multimodal systems, the role of small grains was associated with shielding the coarser ones from fragmentation and delaying subsequent shear localization. Cao et al. (2024) discussed the effect of grain size distribution on the seismogenic potential of basalts in the lunar environment and inferred that gouges with a small mean grain size facilitate fault instability and strength recovery. Bedford and Faulkner (2021) proposed that fine-grained quartz gouge and high normal stresses promoted unstable stick-slip behavior, primarily localized at Y-shear planes parallel to shearing direction. Rattez et al. (2021) partially addressed some of the grain size effects on the frictional behavior and stability of simulated carbonate fault gouge, quantified using rate-and-state friction parameters (RSF) (Dieterich, 1972, 1979, 1981; Marone, 1998; Ruina, 1983). They observed significant fault weakening in experimental samples and interpreted their findings to be due to the dissolution of the finest calcite particles. However, their approach utilized a different configuration of the conventional rotary shear apparatus (rheometer) where shear stress was solely applied along the inner circle and normal stress on both the inner and outer ring, in opposite directions. Hence, the applied normal stresses were potentially underestimated.

Until now, typical geothermal developments were the ones targeting matrix-dominated flow reservoirs originating mainly from siliciclastic sedimentary systems (i.e., sandstones) and also from some carbonate systems. To allow for energy utilization beyond district heating, the exploitation of deeper systems would become necessary. Going deeper would imply that matrix permeability would decrease, and fracture and fault permeability would subsequently become increasingly important. For Northwest Europe, the majority of deep formations are carbonate rocks. The Upper Jurassic (Malm) formation is a geothermal reservoir in the Molasse Basin in Southern Germany, exhibiting mixed matrix and fracture permeability together (Schintgen & Moeck, 2021). The Lower Carboniferous Dinantian formation is a geothermal reservoir in the Netherlands and Belgium, with fractures and/or faults contributing to the geothermal fluid flow (Broothaers et al., 2019, 2021; Kane, Naderloo, et al., 2025). The Californië geothermal wells were the first ones in the Netherlands to target the Dinantian formation, which is also the main deep geothermal target considered in the national geothermal energy development strategy (Heijnen et al., 2019). Belgium has already been exploiting geothermal energy from the same formation with the Balmatt geothermal project, operated by VITO (S. Bos & Laenen, 2017).

Notably, the exploitation of faulted and fractured carbonate reservoirs for geothermal energy has gained traction across Europe, coinciding with an increase in reported cases of triggered seismicity. In the past years, seismic activity has been recorded close to the aforementioned geothermal operations, and both Californië (2018) and Balmatt (2023) projects were shut down. For the Californië project, Buijze et al. (2019), Vörös and Baisch (2022) inferred that the mechanism of thermal contraction provided a possible explanation for the observed induced seismicity. However, they also highlighted that the chemical effects on fault strength and reactivation potential in this specific field have not been studied. The Balmatt doublet also ceased production after receiving a red light on the traffic light system due to a recorded seismic response (Kinscher et al., 2023). In the Molasse Basin, Germany, seismic activity was recorded in the operated areas, though not to an extent that led to projects shutting down. The interpretation of the earthquakes in this region suggested that reactivation of pre-existing faults only occurred after significant thermal and chemical effects (Seithel et al., 2019). However, no specifics were mentioned. In addition, natural seismicity originating from carbonate-bearing faults has been extensively documented, including events such as the 1995 Aigion earthquake in Greece (Bernard et al., 1997), the 2008 Wenchuan earthquake in China (Budach et al., 2018), and several notable seismic sequences in Italy, such as the 2009 L'Aquila earthquake (Valoroso et al., 2014) and the 2016–2017 Amatrice–Norcia events (Pizzi et al., 2017). Both natural and induced seismicity highlight the importance of investigating coupled hydro-chemo-mechanical processes that govern fault reactivation and seismic potential in carbonate formations.

Despite increased interest, the temporal and spatial heterogeneity of fault-zone properties and their dynamic response to natural tectonic processes or human intervention remain poorly understood. The projected increase in exploitation of fractured and faulted carbonate geothermal reservoirs necessitates the improved understanding of the temporal evolution of fault strength before, during, and after seismicity, particularly focusing on fluid-rock chemical interactions. This would lead to more effective risk management strategies with respect to induced seismicity triggered by fluid-rock interactions in carbonate gouge-filled faulted media. However, downscaling the effect from nature to the laboratory poses the challenge of capturing the chemical effects on the fault strength in the laboratory timescale context. This calls for a systematic investigation of the effect of grain size on carbonate friction, which would allow us to better distinguish between the effect of chemical reactions and grain size distribution itself. In this study, we will focus on the effect of grain size distribution and the presence of different fluids during reactivation, frictional healing, and relaxation of simulated carbonate mature faults.

2. Materials and Methods

2.1. Starting Material

2.1.1. Simulated Fault Gouge

We obtained the simulated carbonate gouge from material from the Leffe Quarry in Dinant, Belgium. The quarry exposes formations of Viséan, Lower Carboniferous age (Mozafari et al., 2019; Poty, 2016). The outcrop material was crushed with a ball mill and sieved to obtain different grain size fractions to simulate natural fault wear material (fault gouge) typically found in the principal slip zones in faults (Engelder, 1974). Thermo-Gravimetric Analysis showed the presence of CaCO_3 in the sample solely.

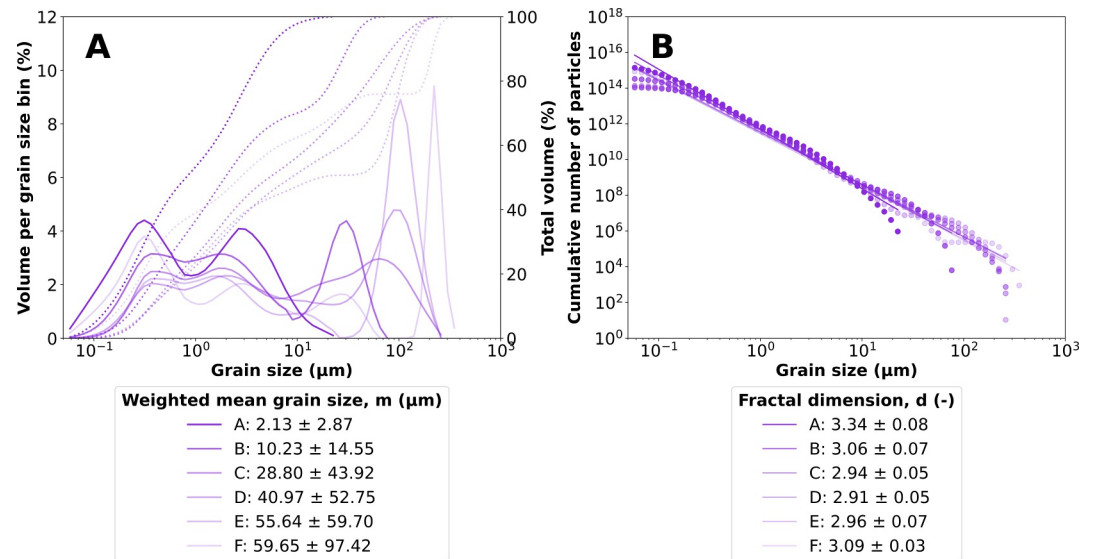


Figure 1. (a) Initial grain size distribution of the different samples used for the experiments. (b) The number of particles with the corresponding grain size of the initial material. We get the fractal dimension, d , from the slope of the linear fit in plot (b).

We created the starting material by crushing and subsequently sieving it with sieve sizes 212, 125, and 45 μm grid size to create different grain size distributions (Figure 1a and Table 1). We used a Laser particle sizer Malvern Mastersizer S to measure the grain size distribution presented in Figure 1a. We conducted at least 4 measurements for the samples A-E and 1 measurement for sample F. We created an additional sample batch of <10 μm by crushing in an MCrone XRD micro-mill in the presence of ethanol for 5 min.

One of the ways to evaluate whether the simulated fault gouge was comparable to natural fault gouge was by assessing the fractal dimension of the grain size distribution. The fractal dimension (d) was defined by $N = R^d$, where N was the number of gouge particles of diameter R . Sammis et al. (1987) reported that the fractal dimension of natural fault gouge retrieved from a two-dimensional cross-section was in the range of 1.18–1.45. In a three-dimensional volume, it scaled to more than 2.18–2.45, as a general result of the fractal theory, which states that the fractal dimension of an isotropic three-dimensional figure is greater by unity compared to a two-dimensional cross-section of it. Biegel et al. (1989), Steacy and Sammis (1991), An and Sammis (1994), Storti et al. (2003) have reported values of fractal dimension between 2 and 3, which occasionally exceeded 3, for extremely pulverized or fine-grained natural fault gouges. We calculated the fractal dimensions of the simulated fault gouge and found values similar to the literature mentioned above (Figure 1b). We considered the effect of undersampling the fine and larger grain sizes, which did not significantly impact the fractal dimension. Note that through this paper, we grouped experiments based on their mean grain size and fractal dimension as explained in the first column of Table 1, and identify them with Exp. ID: A, B, etc., for brevity.

Table 1

Statistics of Grain Size Analyses Along With the Corresponding Fractal Dimension Calculated From the Linear Fit of Particle Number and Grain Size

Exp. ID	Sieve size	Mean, μ_{gs} (μm)	Standard deviation, sd_{gs} (μm)	Fractal dimension, d_{gs} (-)	Standard deviation, sd_d (-)
A	<10	2.13	2.87	3.34	0.08
B	<45	10.23	14.55	3.06	0.07
C	<45 _{50%} , <125 _{50%}	28.80	43.92	2.94	0.05
D	<125	40.97	52.75	2.91	0.05
E	45–125	55.64	59.70	2.96	0.07
F	125–212	59.65	97.41	3.09	0.03

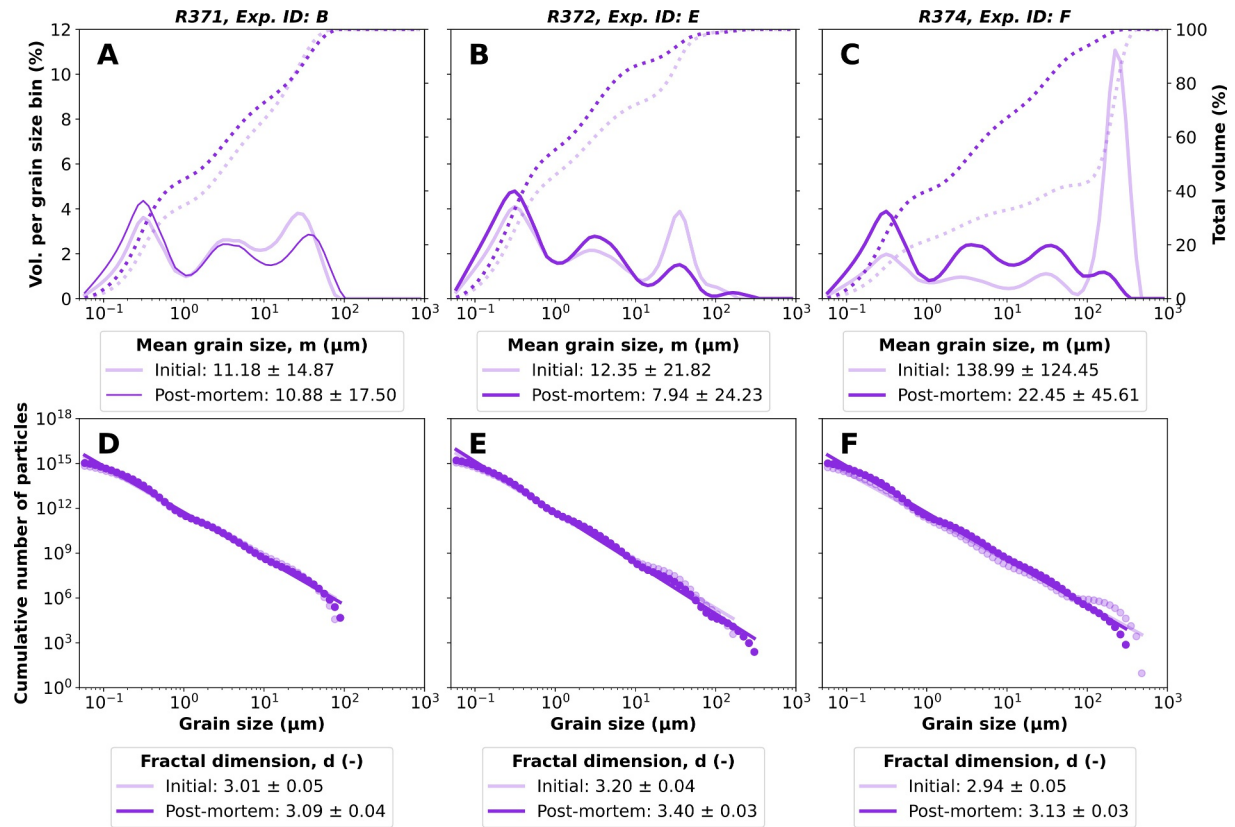


Figure 2. Initial and post-mortem grain size distribution of the samples that experience all normal stress steps but shearing only at the maturation phase (S2), with their corresponding mean grain size (a–c) and their particle number plots with annotated fractal dimension on each distribution (d–f).

2.1.2. Fluid Chemistry

We conducted the experiments in three conditions: air dry with a constant humidity between 50% and 80%, depending on the seasonal conditions, recorded in the lab, saturated with de-ionized (DI) water, and with a geothermal brine obtained from VITO, extracted from the production well of the geothermal site in Mol, Belgium, operating in the Dinantian formation. S. Bos and Laenen (2017) presented the downhole chemical composition of the brine that we used for our experiments, which showed the brine was rich in Na^+ (49.6–49.8 g/L) and Cl^- (98.1–100.2 g/L) ions.

2.2. Experimental Apparatus and Protocol

We conducted experiments on simulated fault gouge using the Rotary Shear Apparatus (RAP) developed and installed at Utrecht University and described by Arts et al. (2024), Korkolis et al. (2021). The sample assembly comprised two grooved piston rings with an inner and outer radius (r_1 and r_2) of 35 mm and 50 mm, respectively. We calculated the imposed velocities and displacements to the mean radius (r_m) of the sample (42.5 mm). We compressed and leveled all samples manually (0.1 mm variability in thickness) before emplacement in the rotary-shear apparatus. The 0.7 mm deep grooves gripped onto the initial approximately 3.5 mm thick gouge layer. Two pressure transducers (PT) were installed in the lower piston to measure local pore pressures in wet experiments. The sample was radially confined by a tightly fitted inner and outer brass ring and sealed with O-rings. Two fluid ports on opposite sides of the outer ring allowed for fluid injection and drainage of the sample. An ISCO pump was connected to one of the fluid ports to saturate the sample with DI water. We recorded and acquired the mechanical and pore pressure data at 50 Hz and 16-bit resolution, using an Elsys TraNET EPC. All of the resolution values reported here have been calculated with the respective transducer(s) at constant load or constant position, at steady-state conditions.

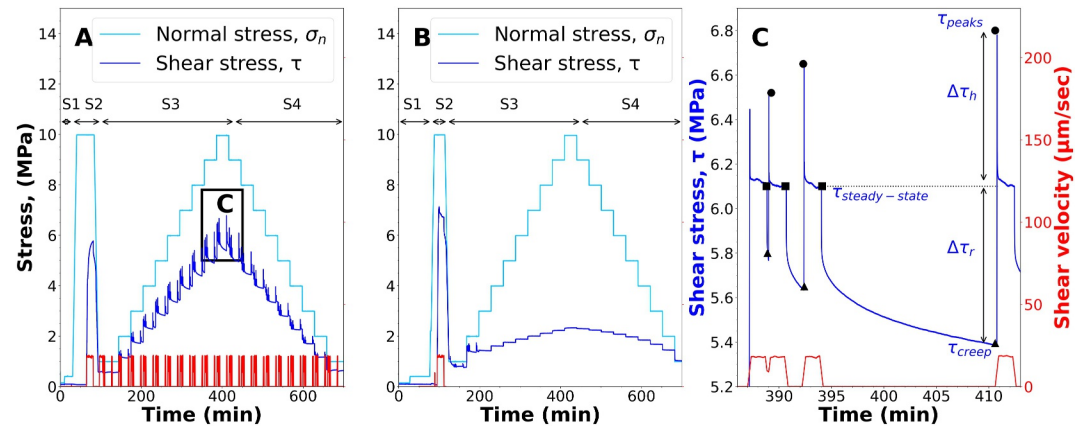


Figure 3. Example protocol showing how normal stress and shear velocity are changed during an experiment. (a) We extract mechanical data for the different stages S1, S2, S3, and S4. (b) The protocol for the reference experiments is to determine how the grain size distribution is affected by the two run-in phases, S1 and S2. (c) Detailed description of different shear stress values during a slide-hold-slide and normal stress step.

We conducted two types of experiments, and the protocol involved the following steps, represented in Figures 3a and 3b. The first type of experiments included steps S1–S4, and the second one omitted the shearing (slide-hold-slide) of steps S3 and S4:

1. *Saturation (S1)*: We applied a small normal stress of 0.4 MPa before saturating the sample. We saturated using the corresponding fluid (DI water or brine) with 500 kPa of pore fluid pressure using an ISCO pump in pressure control. We stopped the ISCO pump when we observed droplets from the outlet annulus, indicating that saturation was achieved.
2. *Maturation-compaction (S2)*: We sheared the simulated gouge at 10 MPa of normal stress, with a constant shearing velocity of $20 \frac{\mu\text{m}}{\text{s}}$ for 1000 s equal to 20 mm of displacement. We used this run-in phase to produce a mature microstructure and minimize grain size reduction effects by cataclasis during the slide-hold-slides in subsequent upsteps and downsteps in normal stress.
3. *Upsteps (S3)*: We imposed a range of normal stresses of 1–10 MPa on the sample with a 1 MPa interval. Every step included a slide-hold-slide protocol with hold times 10–100–1000 s and a constant shearing velocity of $20 \frac{\mu\text{m}}{\text{s}}$ for 100 s equal to 2 mm of displacement. We imposed a 10 mm displacement (duration = 500 s) at the 1 MPa step before the first 10 s hold time. For the second type of experiments, we omitted the shearing procedure.
4. *Downsteps (S4)*: We step-wise decreased the normal stress from 10 to 1 MPa with a 1 MPa interval. We imposed the slide-hold-slide protocol similar to the previous phase. Similarly, here we skipped the slide-hold-slide for the second type of experiments.

We measured the initial and post-mortem (i.e., after 20 mm of sliding) grain size distribution of each reference experiment with the laser particle size analyzer. The results are described in Figure 2.

We conducted all wet experiments under drained conditions, meaning the outlet was open to drain the fluid out of the assembly, and the fluid pump was stopped. After the experiment, we carefully collected the wet samples and placed them in the oven to evaporate the fluid at 50°C for 12 hr. We saturated the dried samples in epoxy and created thin sections, cut approximately parallel to the shearing direction. We conducted SEM analysis using a Gemini SEM (BSE/EDX) in back-scatter mode. The overview of the experiments is in Table 2.

2.3. Data Acquisition and Processing

2.3.1. Raw Data Processing

Throughout the experiments, we logged axial load, axial displacement, rotational displacement, torque, and local pore fluid pressure at a frequency of 50 Hz. We processed the raw data to obtain shear stress, τ (Pa) (Equation 1), normal stress, σ_n (Pa) (Equation 2), apparent friction coefficient μ (–) assuming that cohesion equals zero

Table 2
Table of Experiments With Their Corresponding Grain Size and Shearing History

ID	Fluid	Sieve cluster (μm)	Mean grain size (μm)	Fractal dimension, d (—)	Phases of shearing
R334	Air dry	gs < 45	10.23	2.25	S2, S3, S4
R303	Air dry	gs < 125	40.97	2.19	S2, S3, S4
R305	Air dry	gs < 125	40.97	2.19	S2, S3, S4
R314	Air dry	gs < 125	40.97	2.19	S2, S3, S4
R347	Air dry	45 < gs < 125	55.63	2.24	S2, S3, S4
R428	DI water	gs < 10	2.12	2.25	S2, S3, S4
R431	DI water	gs < 10	2.12	2.25	S2, S3, S4
R375	DI water	gs < 45	10.23	2.25	S2, S3, S4
R383	DI water	gs < 45	10.23	2.25	S2, S3, S4
R335	DI water	50% < 45, 50% < 125	28.80	2.18	S2, S3, S4
R307	DI water	gs < 125	40.97	2.19	S2, S3, S4
R323	DI water	gs < 125	40.97	2.19	S2, S3, S4
R337	DI water	45 < gs < 125	55.63	2.24	S2, S3, S4
R376	DI water	45 < gs < 125	55.63	2.24	S2, S3, S4
R384	DI water	125 < gs < 212	59.64	2.45	S2, S3, S4
R387	DI water	125 < gs < 212	59.64	2.45	S2, S3, S4
R371	DI water	gs < 45	10.23	2.25	S2
R372	DI water	45 < gs < 125	55.63	2.24	S2
R374	DI water	125 < gs < 212	59.64	2.45	S2
R386	Brine	gs < 45	10.23	2.25	S2, S3, S4
R385	Brine	45 < gs < 125	55.63	2.24	S2, S3, S4

(Equation 3), and shear displacement d (mm). f_s ($N \cdot m$) is the torque, f_n (kN) is the normal force, r_m (m) is the mean sample radius and A (m^2) is the sample surface.

$$\tau = \frac{f_s}{A * r_m} \quad (1)$$

$$\sigma_n = \frac{f_n}{A} \quad (2)$$

$$\mu = \frac{\tau}{\sigma_n} \quad (3)$$

We used the shear stress time series to define the peak shear stress (τ_{peaks}) at t_0 , t_{10} , t_{100} , t_{1000} , creep shear stress (τ_{creep}) at t_{10} , t_{100} , t_{1000} and steady-state shear stress ($\tau_{steady-state}$) at t_0 , t_{10} , t_{100} , t_{1000} (Figure 3c). We selected the first peak shear stress at the beginning of each reshearing, the creep shear stress at the end of each hold period, and the steady-state shear stress at the end of each slide stage (Figure 3c). We repeated this process for every normal stress step, for upsteps, and for downsteps.

2.3.2. Mohr-Coulomb Reactivation Envelopes

The Mohr-Coulomb failure criterion (J. Jaeger, 1971; J. C. Jaeger et al., 2009; R. H. Sibson, 1986) has been widely adopted to describe fault strength (Coulomb, 1776; Mohr, 1900; R. H. Sibson, 1990; R. Sibson, 1992). When the term of the cohesion in Equation 4 is zero (which is not always true, especially when clay minerals are present (Ikari & Kopf, 2011)), the Mohr-Coulomb failure criterion reduces to Amontons' law of friction (King Hubbert & Rubey, 1959; Lapusta & Barbot, 2012; Scholz, 2019).

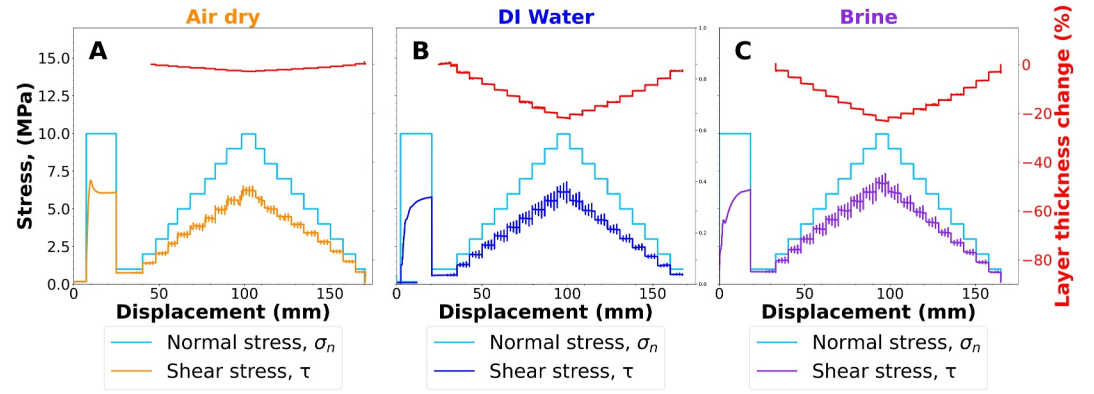


Figure 4. Typical results showing shear stress as a function of shear displacement for (a) air dry samples; (b) samples saturated with DI water, and (c) brine-saturated samples, including layer thickness as a function of shear displacement. All experiments are performed on the sample batch with ID: B, which corresponds to a mean grain size of 10.23 μm .

$$\tau = c(\pm\epsilon_c) + \mu_{int}(\pm\epsilon_\mu) * (\sigma_n) \quad (4)$$

where τ is the shear strength, σ_n is the normal stress, c is the intercept, μ_{int} is the slope or coefficient of internal friction. We included as well the standard error of the intercept (ϵ_c) and slope (ϵ_μ) obtained from the linear fit of the data.

2.3.3. Healing, Healing Rate, Relaxation, and Relaxation Rate

We defined several strength parameters to describe the strength evolution during and after hold periods. These were the healing ($\Delta\mu_h$) and relaxation ($\Delta\mu_r$) values. Here, healing was defined as the difference between the $\mu_{int(peak)}$, at every healing time step (Equation 5). Relaxation was defined as the difference between the $\mu_{int(steady-state)}$ and $\mu_{int(creep)}$ (Equation 6), both logged at the same load-point displacement.

$$\Delta\mu_h = \mu_{int(peak)} - \mu_{int(steady-state)} \quad (5)$$

$$\Delta\mu_r = \mu_{int(steady-state)} - \mu_{int(creep)} \quad (6)$$

A healing/relaxation-rate-per-decade β (Beeler et al., 1994; Karner & Marone, 1998; Marone & Scholz, 1989; Nakatani & Scholz, 2004; Niemeijer et al., 2008; Pluymakers & Niemeijer, 2015; Yasuhara et al., 2005) was constrained by a log-linear least squares regression of healing ($\Delta\mu_h$) and relaxation ($\Delta\mu_r$) versus hold time (t). We included the accumulated errors occurring from the data processing based on Equation 7.

$$\epsilon_{\beta\Delta\mu_{h/r}} = \epsilon_{\mu_{(t/h)}} + \epsilon_{\Delta\mu_{(t/h)}} \quad (7)$$

3. Results

3.1. Initial Observations

In Figure 4, we show a typical plot of the evolution of shear stress and layer thickness as a response to shear velocity and normal stress stepping for three different experiments at different fluid conditions for samples with a mean grain size of 10.23 μm (Exp. ID: B). Shear stress increased at every normal stress step for all experiments. Wet experiments displayed higher shear stress values compared to air dry experiments. Moreover, wet experiments exhibited a more prominent layer thickness decrease compared to the air dry ones, for every normal stress step. The resolution of the data set was not sufficient to capture layer thickness fluctuation during the slide-hold-slide protocol at every normal stress step.

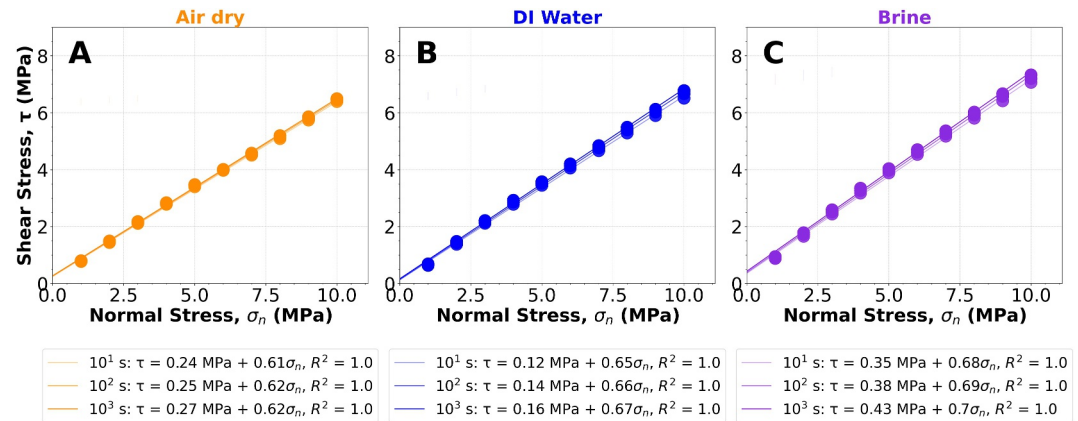


Figure 5. Examples of the Mohr-Coulomb reactivation envelopes associated with peak shear stresses for (a) air dry samples, (b) samples saturated with DI water, and (c) brine-saturated samples. All experiments shown here are performed on the sample batch with ID: B, which corresponds to a mean grain size of 10.23 μm .

3.2. Mohr-Coulomb Reactivation Envelopes

We determined the three types of shear stress values at the given normal stress upsteps and downsteps as described in the methodology. We utilized a linear Mohr-Coulomb fault failure criterion as described in Equation 4. The standard errors associated with the linear regression were noted within a 95% confidence interval.

We demonstrate an example of the reactivation envelopes for every hold time for experiment ID: B (mean grain size of 10.23 μm) (Figure 5). All data showed an excellent linear fit with a goodness of fit $R^2 > 0.99$. It is noteworthy that the intercepts of all linear fits were close to zero within the error bars, regardless of hold time, indicating that no or limited cohesion was developed. To further investigate the effect of the presence or absence of fluids and hold time on the envelopes, we only analyzed the coefficient of internal friction.

For prolonged holding time, the coefficient of internal friction originating from the peak and steady-state shear stress did not exhibit any difference between air dry and wet conditions (Figures 6a–6f). However, the coefficient of internal friction associated with the creep shear stress was lower for air dry conditions compared to wet (Figures 6g–6i). In all cases, there was no discernible difference between DI water and the brine.

For the air dry experiments, only the coefficient of internal friction originating from peak and steady-state shear stress exhibited a positive correlation with increasing mean grain size of the starting material. The coefficient of internal friction originating from creep shear stress was grain-size independent. All wet experiments with DI water exhibited a positive correlation with increasing grain size. There were insufficient data on experiments with brine to infer any grain size dependence of the coefficient of internal friction.

3.3. Healing and Relaxation Absolute Values and Rates

Wet experiments demonstrated higher values of healing (Figures 7a–7c) and relaxation (Figures 7d–7f) than air dry ones. We did not observe any clear trend in healing with different mean of grain size. Experiments with smaller mean grain size in the presence of DI water and brine exhibited lower relaxation values compared to the bigger initial grain size, both on upsteps and downsteps. However, we observed a large uncertainty beyond the mentioned trends in all experiments.

All wet experiments exhibited rates of relaxation and healing rates, within the same range. In general, wet experiments demonstrated higher healing rate and relaxation rate values than air dry ones (Figure 8). However, since there were only 3 hold periods, there was significant uncertainty in the log-linear fit. We observed no grain size dependence of healing rate and relaxation rate in air dry experiments. For wet experiments, within the uncertainty range, grain size distribution had no systematic effect on the healing rate, though initial mean grain size positively correlated with relaxation rates in DI water-saturated experiments.

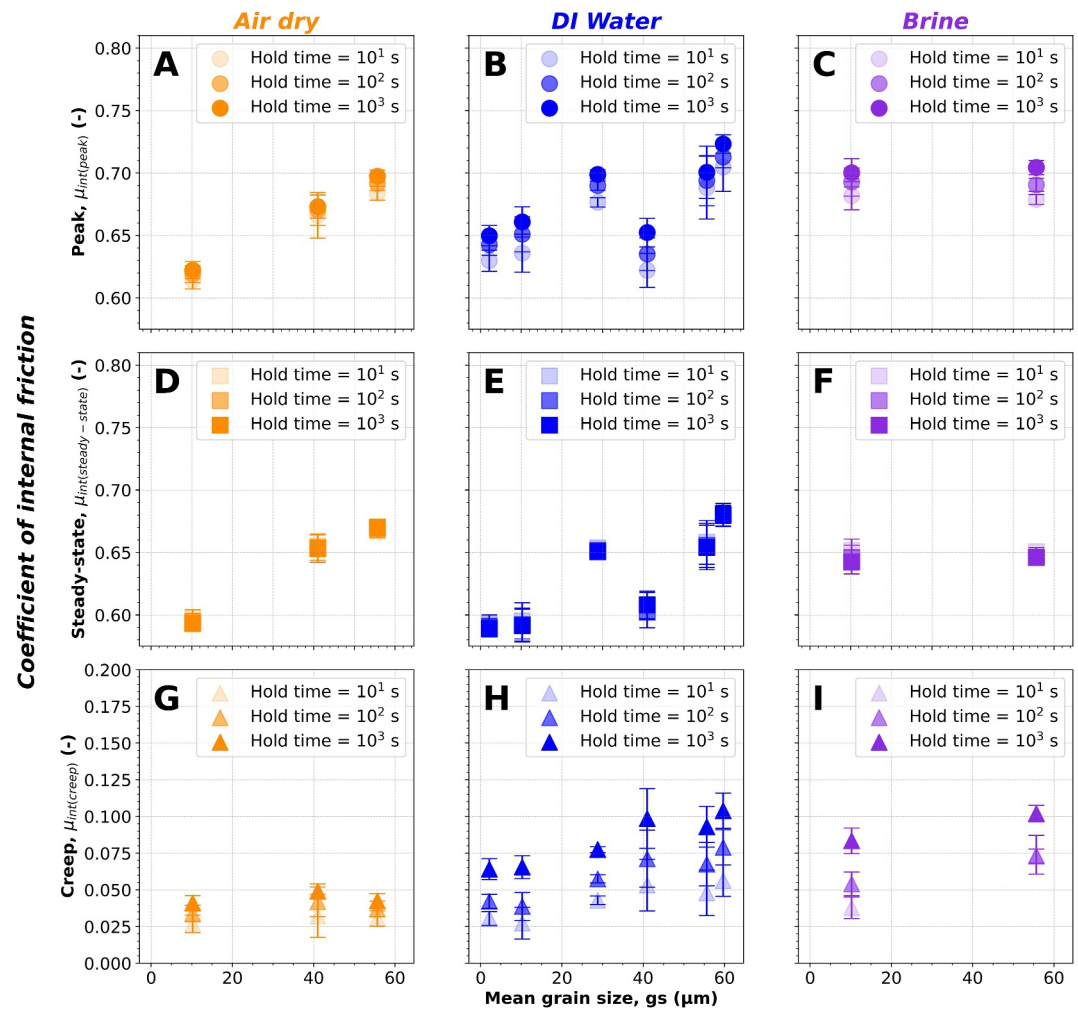


Figure 6. Results of coefficient of internal friction of Mohr-Coulomb envelopes resulting from peak shear stress (a–c), steady-state shear stress (d–f), creep shear stress (g–i) and for the different fluids (air dry on the left, DI water in the center, brine on the right).

3.4. Microstructures

3.4.1. Overview

Table 3 summarizes the characteristics of each grain size class with some examples. In brief, we observed that with increasing mean grain size, the bulk gouge did not exhibit any shearing and/or fracture features. The shape of the clasts transitioned from angular to rounded, and matrix porosity increased. Additionally, the thickness of the PSZ increased with increasing mean grain size along with the presence of shear and/or fracture features. Hence, with increasing mean grain size, there is a transition in strain accommodation from the bulk gouge to localized accommodation in the PSZ. Below we present a detailed description of each sample.

3.4.2. Wet Samples Sheared at Stages S2, S3, S4

We showed SEM-BSE images (Figures 9 and 10) of the gouges with different initial grain size distributions (Table 2). We differentiated between a relatively undeformed bulk gouge and a principal slip zone (PSZ), which we assumed accommodated most of the displacement. The PSZ was defined by a zone of reduced grain size and porosity and shear fracture arrays associated with the main shear zone. Additionally, we identified shear fractures (R1, R2, P, T, and Y-shears) following the terminology of Logan (1979) and qualitatively characterized porosity in the bulk gouge as well as the PSZ. We summarized all characteristics observed in all microstructured in Table 3.

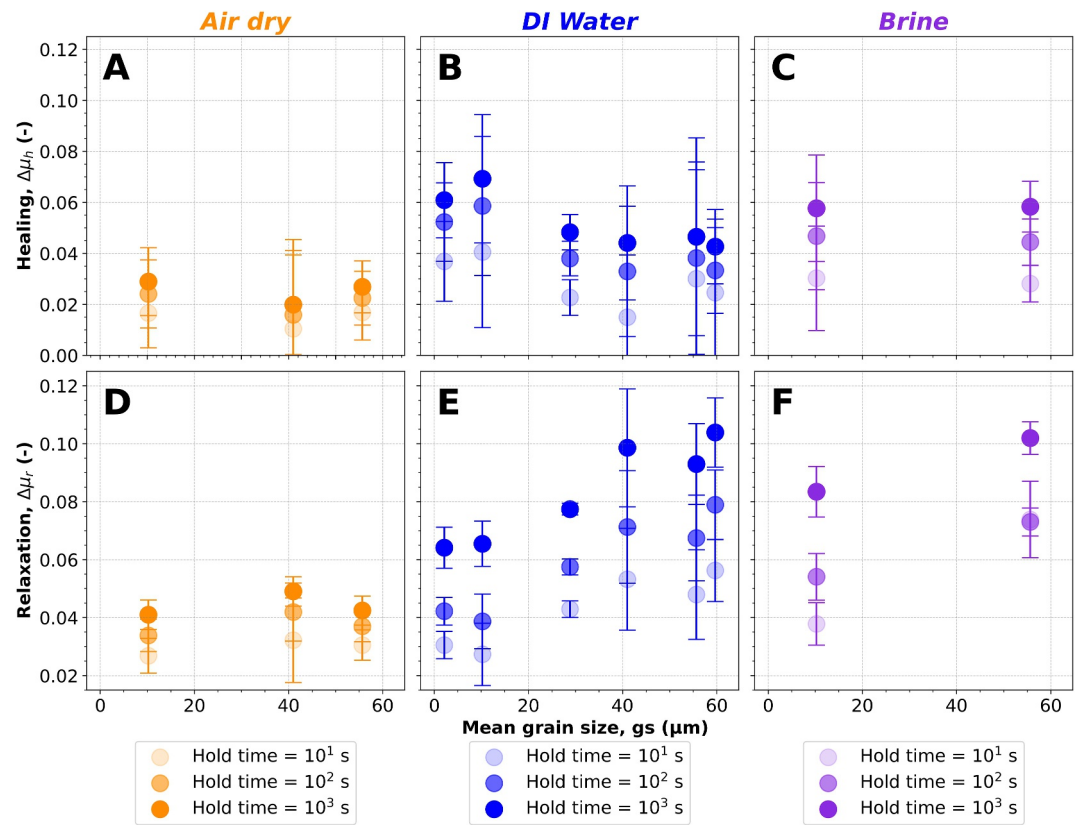


Figure 7. Results of healing (a–c) and relaxation (d–f) for the different fluids (air dry on the left, DI water in the center, brine on the right). Note that the vertical axes are restricted to positive values since this has a physical meaning.

3.4.2.1. Mean Grain Size $2.12 \mu m$

The bulk gouge predominantly contained a fine-grained matrix of micro to nanoscale fragments (Figure 9a). We did not observe a PSZ either because it was absent or we did not recover it. The R1 fractures showed a regular spacing of 2–3 mm and often terminated at the bottom of a tooth at the stationary side. They were oblique to the shearing orientation and characterized by slightly higher porosities than the matrix of the gouge. The imprint of the piston teeth was missing from the rotary side, where most of the R1 fractures flattened out, becoming almost parallel to the shear. In the remainder of the sample, the angle of the R1 fractures remained rather constant,

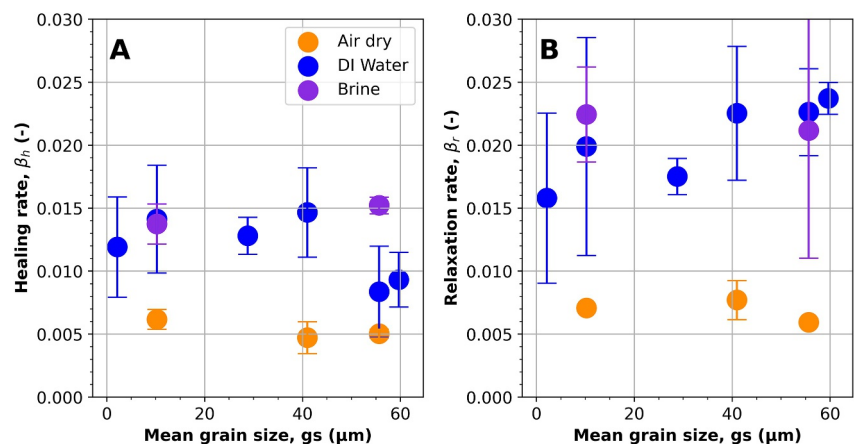


Figure 8. Results of healing (a) and relaxation (b) rate for the different fluids (air dry, DI water, brine).

Table 3*Observations Derived From Microstructures in Figures 9 and 10 Regarding the Bulk Gouge and the PSZ*

Shearing stage			S2, S3, S4				S2 (reference experiments)	
Experiment ID			A: R431	B: R375	D: R307	E: R376	F: R384	
Mean grain size (μm)			2.12	10.23	40.97	55.63	59.64	
Observations	Bulk gouge	Clast shape	–	–	Angular	Rounded & angular	Rounded & angular	Rounded & angular
		Fractured clasts	–	–	Yes	Yes	No	No
		Matrix grain size (μm)	<10	<10	<10	<10	<10	<10
		Porosity (%)	<5	<5	<10	<10	<10	<10
		R1	Dominant	Dominant	Dominant	–	–	–
		R2	Present	Present	–	–	–	–
		T	–	Present	–	–	Present	–
		P	–	–	–	–	–	–
		Y	Present	–	–	–	–	–
	Principal Slip Zone (PSZ)	Thickness (μm)	Not recovered	100–200	200–500	~500	1000	~800
		Clast shape	recovered	–	Rounded & angular	Rounded	Rounded	Rounded & angular
		Fractured clasts		–	Yes	No	Yes	No
		Matrix grain size (μm)		–	<10	<10	<10	<10
		Porosity (%)		–	<5	<5	<5	<5
		R1		–	–	–	Present	–
		R2		–	–	Present	Present	–
		T		–	–	Present	Present	–
		P		–	–	Present	–	Present
		Y		–	–	Present	–	Present

Note. Regular experiments sheared at stages S2, S3, S4 and reference experiments sheared only during S2. Experiment IDs (Rxxx) identify individual laboratory experiments, while labels A–F denote the investigated gouge grain-size classes. The mean grain size (μm) reported for each experiment corresponds to the initial gouge material used in the shearing tests. Microstructural observations include clast morphology, fracturing, matrix grain size, porosity, and the occurrence of deformation structures within the bulk gouge and PSZ. R1 = synthetic Riedel shears; R2 = antithetic Riedel shears; P = P-shears; T = tension fractures; Y = Y-shears. “–” indicates that the feature was not observed.

although some steps could be observed. R2-type Riedel fractures were less frequent and appeared in conjunction with R1-type. They exhibited an almost perpendicular orientation to R1-type fractures and often served as their connection paths. We observed a short shear fracture parallel to the shear orientation that connected the R1-shear fractures and we interpreted that as a Y-shear.

3.4.2.2. Mean Grain Size 10.23 μm

The sample had a bulk gouge that contained a fine-grained matrix of a nano-to-micro scale (Figure 9b). We recovered only a very thin PSZ ($\approx 10\text{--}20\ \mu\text{m}$). The bulk matrix often separated with R1-type Riedel fractures. The fractures exhibited an increased aperture and therefore porosity. They run to the top boundary but disappeared toward the bottom boundary, or they flattened out. The spacing of R1 fractures was larger than in the previous sample. R2-type Riedel fractures were less frequent and appeared in conjunction with R1-type fractures and served as their connection paths. Oriented perpendicularly to the R1, there was also a tension (T)-type fracture, with a meandering shape and increased aperture.

3.4.2.3. Mean Grain Size 40.97 μm

The bulk gouge predominantly contained clasts with both angular and rounded edges suspended around in a fine-grained matrix (Figure 9c). We observed the development of a $\approx 20\text{--}50\ \mu\text{m}$ thin but well-defined PSZ (Figure 9d),

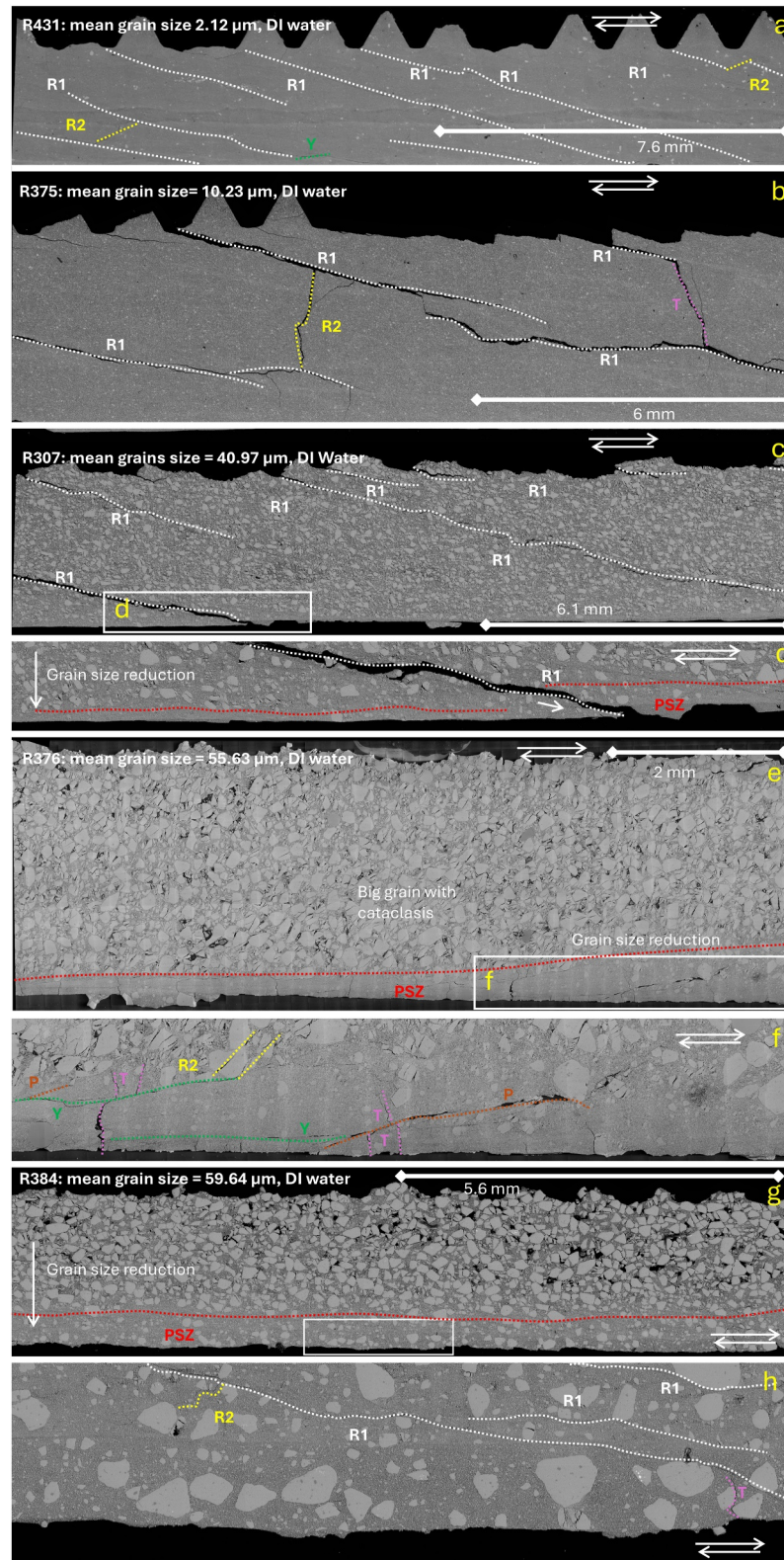


Figure 9. Shearing and fracture features in the bulk gouge and principal slip zone of all experiments that have been sheared at all stages. (a) Mean grain size $2.12 \mu\text{m}$. (b) Mean grain size $10.23 \mu\text{m}$. (c) Mean grain size $40.97 \mu\text{m}$ (d) zoom in the PSZ of (c). (e) Mean grain size $55.63 \mu\text{m}$. (f) zoom in the PSZ of for example) Mean grain size $59.64 \mu\text{m}$. (h) zoom in the PSZ of (g).

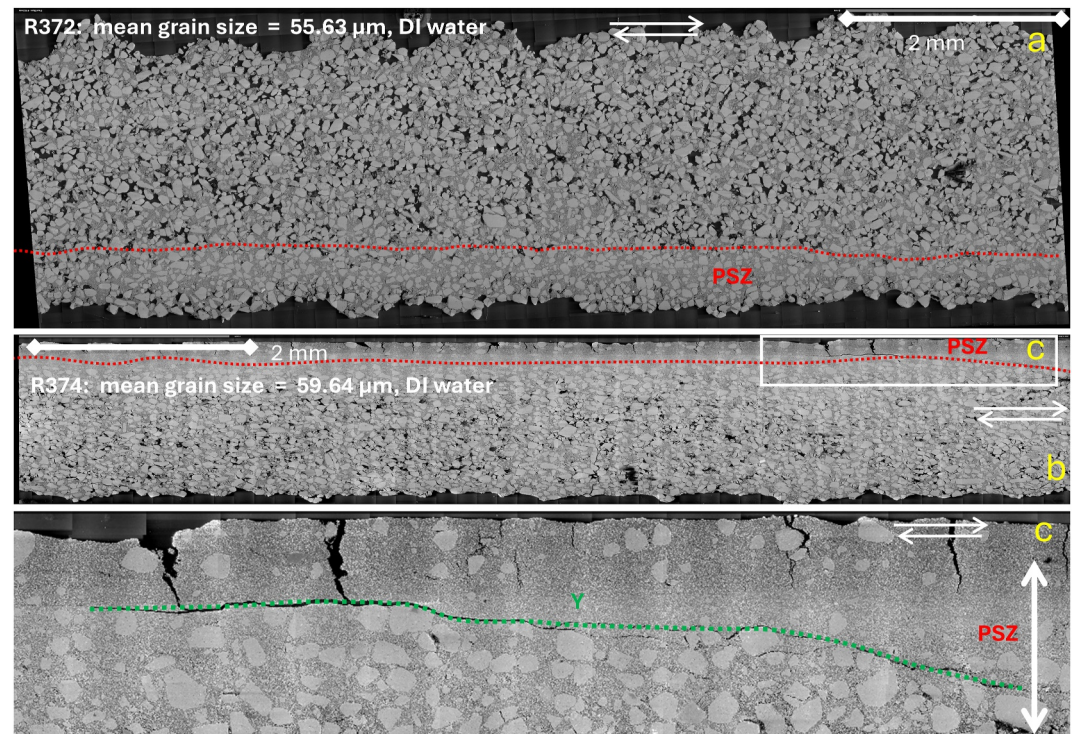


Figure 10. Shearing and fracture features in the bulk gouge and principal slip zone of all experiments that have been sheared only at the maturation (S2) stage. (a) Grain size mean 55.63 μm (b) Grain size mean 59.64 μm . (c) zoom in the PSZ of (b).

characterized by substantially smaller grain size and the absence of large grains and shear features. The bulk matrix of the sample predominantly separated with fractures in an R1-type Riedel shear orientation. They exhibited oblique and meandering orientations to the shearing direction and were characterized by variable aperture. Most R1 fractures terminated and flattened toward the bottom boundary, except for one R1 shear, which cut across the PSZ. Within the bulk gouge, some clasts were intact, whereas others were fractured. When the grains were cracked, they exhibited a crack orientation similar to R1-type fractures.

3.4.2.4. Mean Grain Size 55.63 μm

The sample in Figure 9e had a bulk gouge that included clasts with both angular and rounded edges embedded in a fine-grained matrix. However, the fine-grained matrix preserved porosity defined by the dissolution and/or movement of the margins of the grains. Some clasts were intact, whereas others showed intragranular fractures. When the grains were cracked, qualitatively, they exhibited an almost similar crack orientation. There were no shear features in the matrix. We observed a minimal contribution of Y-oriented fractures with increased aperture but without evidence of a shearing component. We additionally observed a well-defined $\approx 0.5 \text{ mm}$ PSZ (Figure 9f), characterized by substantially smaller grain size (micro-scale) and the presence of a few but well-rounded and uncracked large grains at the lower boundary of the PSZ. The majority of shear features were present in the PSZ. There was an absence of R1-type Riedel shear fractures.

3.4.2.5. Mean Grain Size 59.64 μm

The sample in Figure 9g had a bulk gouge that predominantly contained clasts with both angular and rounded edges embedded in a fine-grained matrix. The clasts away from the PSZ were fractured and angular, whereas the ones closer were intact and rounded. We observed the absence of shear features in the bulk matrix (Figure 9h) and a well-defined and thick PSZ ($\approx 1 \text{ mm}$), characterized by substantially small grain size of the matrix and the presence of large, mainly intact, well-rounded grains. The PSZ exhibited fractures in an R1-type Riedel orientation, with oblique orientations to the shearing direction, a meandering shape, and they terminated at the boundary of PSZ. There was sparse contribution of R2 and tensile (T) fractures that were connected to the R1 fractures.

3.4.3. Reference Wet Samples Sheared at Stage S2

3.4.3.1. Mean Grain Size 55.63 μm

The sample that experienced shearing only at the maturation stage S2 showed a bulk gouge that has both angular and rounded clasts, no intragranular fractures, and a fine matrix with porosity defined by the boundaries for the clasts (Figure 10a). There were no shear features in the matrix. Additionally, we observed a less well-defined PSZ, characterized by substantially more matrix, hence decreased porosity and the presence of uncracked large grains. There were no indicative shearing fractures. It had a relatively stable thickness of 1 mm.

3.4.3.2. Mean Grain Size 59.64 μm

The sample that was sheared only at the maturation stage S2 had a bulk gouge that predominantly contained clasts with both angular and rounded edges embedded in a fine-grained matrix (Figure 10b). The majority of the fragments were intact. The matrix was filled with large voids, which directionally decreased in number toward the transition to the PSZ. This gradual transition marked the PSZ characterized by substantially more matrix material and the presence of large, well-rounded grains (Figure 10c). We detected an elongated Y-shear fracture that define the lower boundary of the PSZ.

4. Discussion

4.1. General Observations

Our results indicated that in air dry samples, the initial mean grain size positively correlated with the coefficient of internal friction of the peak shear stress (Figure 6a), and steady-state shear stress (Figure 6d). Healing and relaxation, in terms of absolute values and rates, remained unaffected (Figures 7 and 8). In wet samples, the initial mean grain size positively correlated with the coefficient of internal friction of the peak shear stress (Figure 6b), steady-state shear stress (Figure 6e), and creep shear stress (Figures 6h and 6i). Healing in terms of absolute values and rates did not exhibit any grain size dependence (Figures 7b, 7c, and 8a), while relaxation absolute values and rates did (Figures 7e, 7f, and 8b).

Additionally, we were able to recover only the wet samples, which remained mechanically intact and sufficiently physically cohesive to allow preparation for microstructural analyses. These analyses revealed that a decrease in initial mean grain size corresponded with a progressively thinner principal slip zone (PSZ), which became indistinct or absent in samples with the initially smallest mean grain size. In the cases where the PSZ was recoverable in fine-grained samples, Riedel shear structures were absent, and we could recover no discernible information on grain size and shape within the PSZ within the resolution of the microstructures. The bulk gouge exhibited frequent Riedel shear features. Conversely, samples with initially larger mean grain size were associated with more frequent development of Riedel shear systems in the PSZ. The bulk gouge had no evidence of Riedel shears and was characterized by more rounded and less fractured clasts.

The evidence indicated pronounced deformation within the PSZ, where grain size and angularity were significantly reduced compared to the initial grain size, resulting in more rounded, fine grains, reflecting localized deformation as well as increased transport distance and energy involved in the mobilization of grains into the shear zones. Solely the microstructures originating from the larger initial mean grain size preserved angular clasts of smaller than 500 nm scale floating around ultra-fine grained gouge.

The bulk gouge of smaller initial mean grain size correlated with qualitatively lower porosity and, where observable, the presence of Riedel shear systems defined by angular and highly fractured clasts. In contrast, samples with a larger initial mean grain size did not exhibit Riedel shear structures; instead, deformation was manifested through grains that were more angular to rounded.

In the following section, we will first discuss the deformation mechanisms active in air dry and wet experiments. We will then elaborate on the effect of initial grain size distribution on the frictional behavior. To conclude, we will unfold the implications of our results for natural seismicity in carbonate terrains, as well as induced seismicity for the operation of geothermal systems in fractured and faulted carbonate reservoirs.

4.2. Comparison of Air Dry and Wet Experiments

In this study, the coefficient of internal friction associated with creep shear stresses (Figure 6g–6i), healing, and relaxation in terms of absolute values and rates (Figures 7 and 8) exhibited higher values for the wet experiments compared to air-dry samples. Additionally, the coefficients of internal friction associated with peak and steady-state shear stresses of air dry samples were comparable to wet samples (Figures 6a–6f).

Pluymakers and Niemeijer (2015) presented results from slide-hold-slide experiments on anhydrite fault gouges, which showed a similar behavior for wet and air dry samples and suggested that humidity in the sample preparation room resulted in water adsorbed to the grain contacts. This residual water allowed water-based deformation mechanisms that need only a nano-film of water to take place, such as pressure solution (Rutter, 1983; Spiers et al., 2004) and intergranular lubrication as was suggested for calcite room-dry gouges, see B. Verberne et al. (2014). Additionally, pressure solution led to porosity reduction, thereby increasing grain contact area (Bos & Spiers, 2002b; B. Bos et al., 2000; Niemeijer & Spiers, 2006) and hence promoting healing and relaxation. Pressure solution also plays a key role in the microphysical-based CNS model for friction of gouge experiments (Chen & Spiers, 2016). Since we observed measurable healing and relaxation in the air dry samples, and humidity between 50% and 80% (season-dependent) in the different laboratory rooms, our results align with this observation, and we therefore speculate that some component of pressure solution was active during deformation.

B. Verberne et al. (2014) proposed that intergranular lubrication arose from water adsorption onto (nano-)grain surfaces. This adsorption process is predominantly governed by the electrokinetic potential of the grain surfaces, which, in the case of calcite, is intricately influenced by the presence of impurities (Cicerone et al., 1992) as well as by pressure and temperature conditions (Rodríguez & Araujo, 2006). Given the high humidity in the laboratory environment, we assume that all grains were coated with a nanofilm of water, which was sufficient to lubricate frictional contact. This explains the similarity in peak and steady-state friction values. Nonetheless, the fully water-wet (or brine-wet) carbonate samples exhibited increased healing and relaxation, which was commonly seen in experiments (Dieterich & Conrad, 1984; Dieterich & Kilgore, 1994; Frye & Marone, 2002). It is well known that the presence of fluids aids grain breakage by decreasing the critical stress needed to propagate grain fracturing (fracture toughness) (Atkinson, 1982; Ovalle et al., 2015; Rice, 1978; Y. Zhang & Buscarnera, 2018). Specifically for calcite, its fracture surface energy is 2–3 times lower in the presence of fluids such as water (Røyne et al., 2011). This reduction reflects the activation of stress corrosion cracking, whereby chemically assisted subcritical crack growth lowers the energy required for bond rupture at crack tips. As a result, the energy needed for carbonate grain failure and hence cataclasis was significantly reduced in wet experiments compared with dry experiments (Atkinson, 1982; Brantut et al., 2013; Rice, 1978). Consequently, cataclasis is expected to be enhanced in wet samples. This interpretation is supported by our thin-section analysis of wet samples, which showed pervasive grain-scale cataclasis in the samples with a mean grain size $>10.23 \mu\text{m}$ in the microstructures. In wet experiments with DI water, bigger grains with round shapes tended to fracture and decompartmentalize into smaller angular components. We located them floating around the finer gouge both at the margins and cores of shear bands (Figure 9). Moreover, once grain size would decrease due to grain fracture, the rate of pressure solution would increase as well (J. Zhang et al., 2010), which in turn would contribute to healing and relaxation absolute values and rates. We thus infer that the reason for increased healing and relaxation in wet experiments compared to dry is that grain cataclasis was enhanced throughout our fluid-saturated experiments, promoting pressure solution in fine grains aided by the presence of fluids. Table 4 summarizes different literature on the frictional properties reported on similar material and comparable experimental conditions, primarily in fluid-saturated conditions.

4.3. Effect of Initial Grain Size Distribution on Frictional Properties

In air dry samples, the coefficient of internal friction of the peak and steady-state shear stresses (Figures 6a–6f) displayed a systematic positive correlation with initial mean grain size. In contrast, the coefficient of internal friction of the creep shear stresses (Figures 6g–6i), the absolute values and rates of healing, and relaxation (Figures 7 and 8) did not. This suggests that in air dry conditions, only peak and syn-sliding shear stress behavior retained sensitivity to grain size, while grain-size-independent mechanisms governed creep, healing, and relaxation. In contrast, under wet conditions, all types of coefficient of internal friction (Figure 6) and relaxation behaviors (in terms of absolute values and rates) (Figures 7d–7f and 8b) exhibited a positive correlation with

Table 4

Summary of Experimental Conditions and Derived Frictional Properties Reported in Previous Studies on Natural and Simulated Carbonate Fault Gouge, Compared With the Results of This Study

Reference	Normal stress (MPa)	Fluid	Shear velocity ($\frac{\mu m}{s}$)	Temperature	Internal friction	Peak friction	Steady-state friction	Creep friction	Healing	Relaxation	Healing rate	Relaxation rate
This study	10	DI water	20	Room	0.6–0.7	0.65–0.73	0.6–0.67	0.025–0.1	0.01–0.07	0.025–0.105	0.003–0.015	0–0.024
Rattez et al. (2021)	1.6	Water, HCL 10%	1.7–33	Room	NA	0.24–0.33	0.14–0.33	NA	NA	NA	NA	NA
B. Carpenter, Collettini, et al. (2016)	10	DI water	10	Room	0.63	NA	0.66–0.67	NA	0.018–0.02	0.048	NA	NA
B. Carpenter et al. (2014)	10	DI water	1–300	Room	NA	NA	0.6	NA	0.01–0.06	0.03–0.11	0.01–0.02	0.035
B. Verberne et al. (2014)	50	DI water	10	Room	NA	0.69–0.72	0.59–0.66	0.002–0.01	NA	NA	NA	NA
Tesei et al. (2014)	10	Hard water	10	Room	NA	NA	0.57–0.70	NA	0.02–0.08	NA	NA	NA
Scuderi et al. (2013)	10	DI water	10	Room	0.63–0.68	0.6–0.7	NA	NA	NA	NA	NA	NA
B. A. Verberne et al. (2010)	58–73	DI water	1.22	Room	NA	NA	0.46–0.71	0.118–0.136	NA	NA	NA	NA

Note. Reported parameters include normal stress, pore fluid, shear velocity, temperature, and frictional metrics such as internal friction, peak friction, steady-state friction, creep friction, healing, and relaxation behavior. Ranges reflect variability across experimental conditions within each study. NA denotes parameters that were not reported or discussed in the original publications.

initial mean grain size, while healing, both in terms of absolute values as well as rates (Figures 7a–7c and 8), remained independent of grain size.

As previously discussed, we interpreted that pressure solution operated within both the bulk gouge and the PSZ and in both air dry and wet samples. Microstructural observations indicated that all samples showed the presence of a PSZ with a finer grain size than the initial mean grain size. The mean grain size and grain shape within the PSZ may be independent of the initial grain size distribution, although we did not have the resolution to confirm this. Therefore, if pressure solution played a role, this might be consistent with a comparable rate of pressure solution in the PSZ for all mean initial grain size distributions of the starting material. This, combined with grain size-independent healing absolute values and rates, suggests that healing occurred within the PSZ.

Additionally, we observed a positive correlation between the initial mean grain size and both the absolute wet relaxation values and rates, as well as PSZ thickness. Relaxation occurred in both the PSZ and bulk gouge and was likely influenced by pressure solution creep (Bos & Spiers, 2002a; Van den Ende & Niemeijer, 2019). The observed thicker PSZ in samples with a larger initial mean grain size implies reduced strain rates within the fine-grained PSZ, which would thereby enhance the relative contribution of pressure solution-controlled creep to the relaxation (Chen & Spiers, 2016; Noël et al., 2024; Van den Ende et al., 2018). We therefore propose that initial grain size distribution primarily influenced PSZ thickness, while PSZ thickness modulated the relaxation behavior.

4.4. Interpretation in the Context of the CNS Microphysical Model

A microphysical interpretation of the observed healing and relaxation behavior in our experiments was complicated by the fact that while the initial grain size was controlled, the subsequent evolution of grain size and microstructure during deformation was not. Microstructural observations indicated that the thickness of the localized boundary-parallel shear zone (PSZ) increased with increasing initial mean grain size. Owing to limited spatial resolution, the final grain size within the PSZ could not be quantified directly. Nonetheless, based on the mechanical data and literature, we inferred in the preceding sections that pressure solution was active during our experiments.

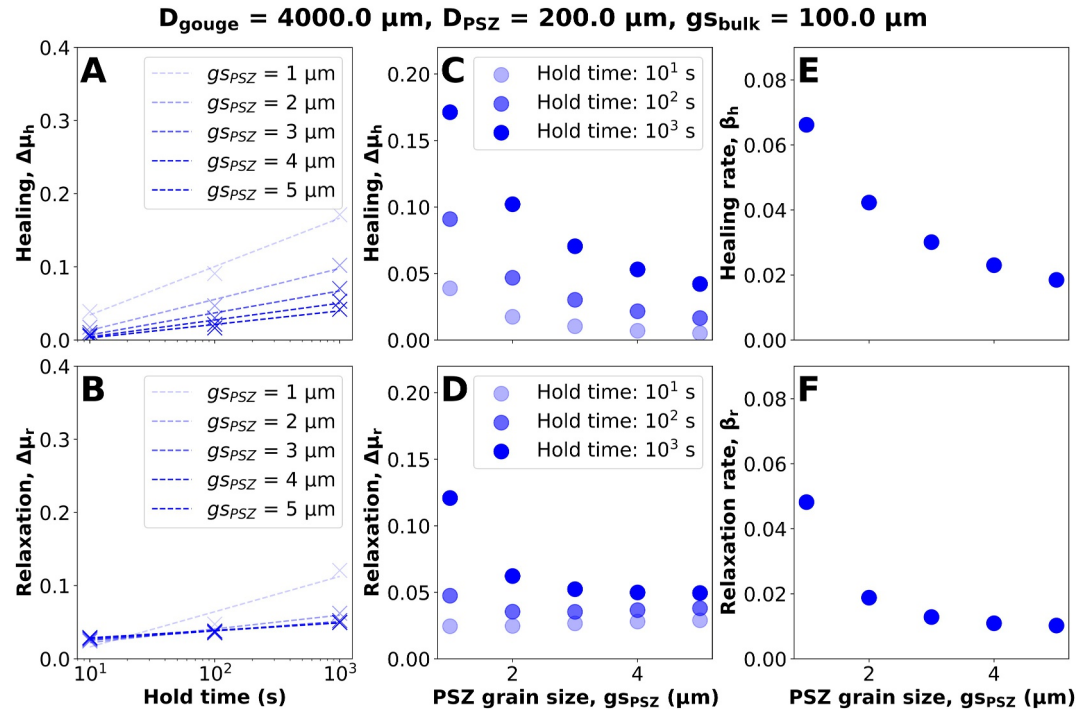


Figure 11. The simulated starting conditions are total gouge thickness: $D_{\text{gouge}} = 4000 \mu\text{m}$, thickness of the Principal Slip Zone (PSZ): $D_{\text{PSZ}} = 200 \mu\text{m}$, and grain size of the bulk gouge: $g_{\text{Sbulk}} = 100 \mu\text{m}$. Effect of Principal Slip Zone (PSZ) grain size on healing and relaxation behavior. (a, b) Log-linear evolution of healing ($\Delta\mu_h = \mu_{\text{peak}} - \mu_{\text{ss}}$) and relaxation ($\Delta\mu_r = \mu_{\text{creep}} - \mu_{\text{ss}}$) as a function of hold time (10–1000 s) for PSZ grain sizes (g_{SPSZ}) between 1 and 5 μm . Dashed lines represent linear regressions in log hold time used to determine healing and relaxation rates (β_h and β_r). (c, d) Dependence of healing and relaxation on PSZ grain size for different hold times (10^1 , 10^2 , and 10^3 s). (e, f) Healing rate (β_h) and relaxation rate (β_r) as a function of PSZ grain size.

To better understand whether this is a plausible explanation, we used the microphysical CNS model from Chen and Spiers (2016) to systematically investigate the influence of key microstructural parameters, namely grain size within the PSZ (Figure 11), grain size in the bulk gouge (Figure 12), and PSZ thickness (Figure 13), on healing and relaxation absolute values and rates. In the CNS framework, frictional strength evolves through porosity evolution, governed by a competition between time-dependent compaction accommodated by pressure solution and slip-dependent dilation associated with frictional sliding and granular flow. A detailed description of the model was provided by Chen and Spiers (2016). We solved the two ordinary differential equations (Equations 33a and 33b) from Chen and Spiers (2016) using ODE solvers (Sundnes, 2023), inserting values corresponding to the experimental protocol of slide-hold-slides at 20 $\mu\text{m/s}$ with holds of 10–100–1000 s. Using otherwise the same parameters as in Chen and Spiers (2016) (i.e., for pure calcite gouge), with solubility and diffusion coefficient adjusted to take into account room temperature. We simulated a slide-hold-slide for each normal stress (1–10 MPa with 1 MPa steps) and extracted the peak, creep, and steady-state shear stress for each simulation. In the subsequent analysis, we followed the methodology of the current study to obtain healing and relaxation absolute values and rates (i.e., to allow comparison between model and experimental results). With the CNS model, we investigated how the grain size in the Principal Slip Zone (PSZ) (g_{SPSZ}), grain size in the bulk (g_{Sbulk}), and thickness of the PSZ (D_{PSZ}) affect healing and relaxation absolute values and rates.

The microphysical model simulations showed that with decreasing grain size within the PSZ, the amount of healing absolute values and rates increased (Figure 11). In contrast, neither changing the bulk grain size (Figure 12) nor the PSZ thickness (Figure 13) affected healing absolute values and rates. The simulations also confirmed that relaxation, in terms of absolute values and rates, was sensitive to all investigated parameters (Figures 11–13). Specifically, smaller grain size promoted increased absolute relaxation values and rates, both within the PSZ (Figure 11) and in the surrounding bulk gouge (Figure 12), and a wider PSZ (Figure 13).

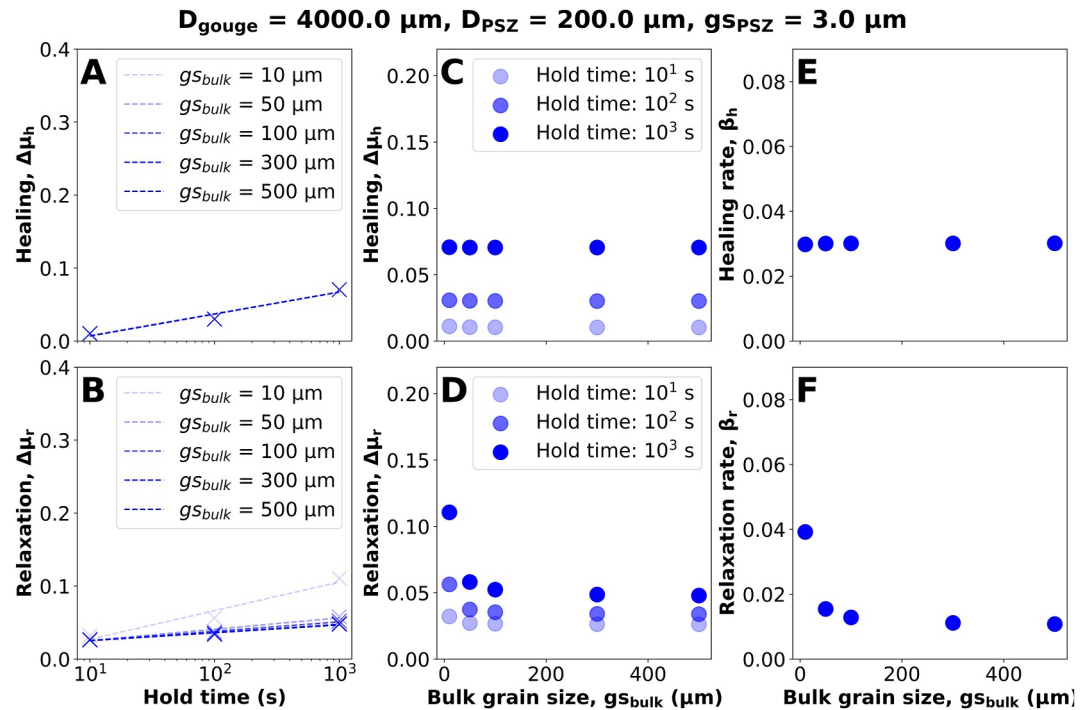


Figure 12. The simulated starting conditions are total gouge thickness: $D_{\text{gouge}} = 4000 \mu\text{m}$, thickness of the Principal Slip Zone (PSZ): $D_{\text{PSZ}} = 200 \mu\text{m}$, and grain size of the Principal Slip Zone: $g_{\text{PSZ}} = 3 \mu\text{m}$. Effect of bulk gouge grain size on healing and relaxation behavior. (a, b) Log-linear evolution of healing ($\Delta\mu_h = \mu_{\text{peak}} - \mu_{\text{ss}}$) and relaxation ($\Delta\mu_r = \mu_{\text{creep}} - \mu_{\text{ss}}$) as a function of hold time (10–1000 s) for bulk grain sizes (g_{bulk}) between 10 and 500 μm . Dashed lines represent linear regressions in log hold time used to determine healing and relaxation rates (β_h and β_r). (c, d) Dependence of healing and relaxation on bulk grain size for different hold times (10^1 , 10^2 , and 10^3 s). (e, f) Healing rate (β_h) and relaxation rate (β_r) as a function of bulk grain size.

Our experimental results with respect to healing absolute values and rates aligned with the trends observed in the microphysical modeling. However, our experiments also showed a decrease in relaxation with decreasing initial grain size, contrary to the CNS-predicted increase. However, the microstructural results (Figure 9) also exhibited a thinner PSZ in the fine-grained experiments. Therefore, the decreased relaxation in terms of absolute values and rates in the experiments could be the result of this thinner PSZ, since PSZ thickness was shown to have a profound effect on healing.

4.5. Implications for Natural and Induced Seismicity and Fault Reactivation in Carbonate Geothermal Reservoirs

Grain cataclasis would shift the mean grain size toward finer particles, hence reducing the mean value of the distribution, regardless of the initial grain size distribution. Our study suggested that such a shift reduced the coefficient of internal friction associated with the amount of shear stress necessary to initiate shearing. Therefore, this would promote fault reactivation and the potential of seismic occurrence. However, since both peak and steady-state internal friction decreased with decreasing grain size (Figures 6a–6f), the stress drop upon the start of sliding would remain similar. Similarly, natural tectonic processes or engineered subsurface interventions for geoelectric applications may involve fluid circulation that may occur in proximity to carbonate mature fault zones. These naturally flowing or injected fluids can promote fluid-rock interaction (e.g., dissolution of granular material) within the fault core and damage zone. The net effect of grain dissolution is an interplay of dissolution of finer grains and reducing the size of the bigger grains, which retains opposing influences and the overall effect depends on the extent of each modification.

Our study additionally suggested that fault healing was likely influenced by pressure solution processes operating within the fine-grained high-strain zones and was largely insensitive to the initial grain size of the material. Given that pressure solution required the presence of fluids, its activity implied that healing in these zones was fluid-

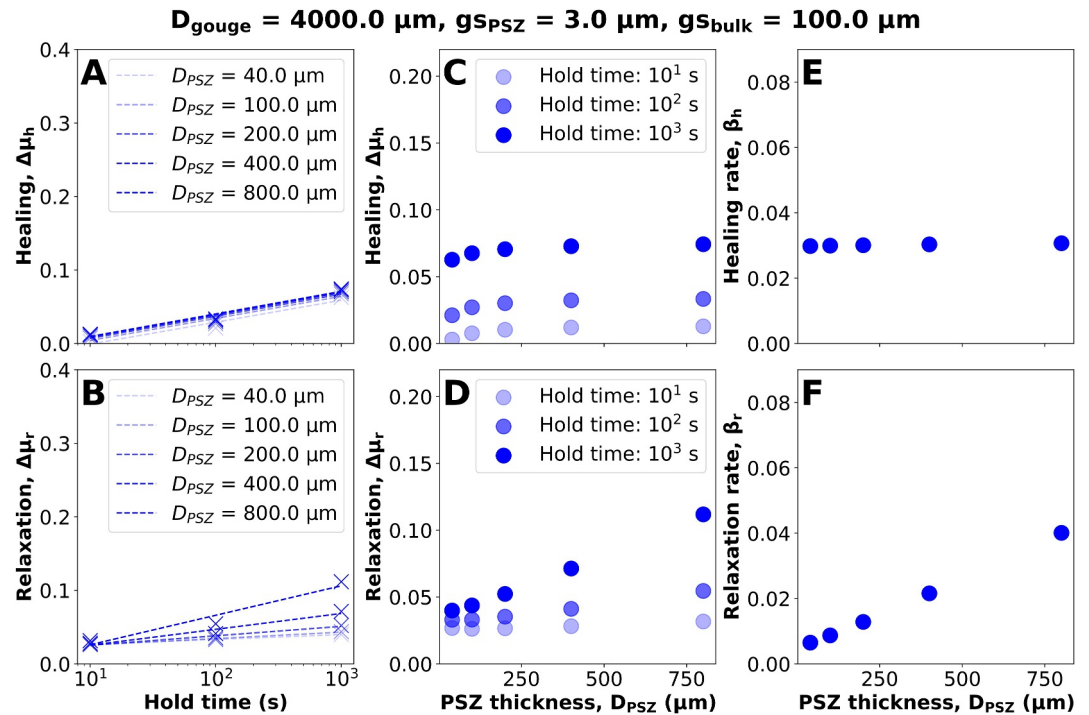


Figure 13. The simulated starting conditions are total gouge thickness: $D_{\text{gouge}} = 4000 \mu\text{m}$, PSZ grain size: $g_{\text{PSZ}} = 3 \mu\text{m}$, and bulk gouge grain size: $g_{\text{bulk}} = 100 \mu\text{m}$. Effect of Principal Slip Zone (PSZ) thickness on healing and relaxation behavior. (a, b) Log-linear evolution of healing ($\Delta\mu_h = \mu_{\text{peak}} - \mu_{\text{ss}}$) and relaxation ($\Delta\mu_r = \mu_{\text{creep}} - \mu_{\text{ss}}$) as a function of hold time (10–1000 s) for PSZ thicknesses (D_{PSZ}) between 40 and 800 μm . Dashed lines represent linear regressions in log hold time used to determine healing and relaxation rates (β_h and β_r). (c, d) Dependence of healing and relaxation on PSZ thickness for different hold times (10^1 , 10^2 , and 10^3 s). (e, f) Healing rate (β_h) and relaxation rate (β_r) as a function of PSZ thickness (D_{PSZ}).

assisted. Consequently, neither fault slip nor fluid–rock interactions and their subsequent grain size modifications were expected to significantly influence the extent or magnitude of healing within the localized deformation regions, unless the grain size (distribution) within the PSZ was affected.

Torabi et al. (2019) reported a positive power-law correlation between carbonate fault core thickness and total fault displacement. Our results suggested that a reduction in the mean grain size within the fault zone, which serves as a proxy for fault displacement, tended to decrease the thickness of high-strain regions. In particular, zones of elevated strain, where slip was predominantly localized, became increasingly narrow relative to the surrounding low-strain matrix. Subsequently, our results focusing on small-scale do not agree with the observations of natural, large-scale faults. This discrepancy may be attributed to the generation of further wear material during fault slip, which can accumulate in the fault core and effectively contribute to fault zone thickening. It is important to note that in our laboratory experiments, there was no influx of granular material during shear. As a result, progressive material production observed in natural fault systems cannot be replicated under our current experimental conditions. Should dissolution of fault material occur from natural or engineered processes, preferentially remove the finer fraction of the grain size distribution, the resulting increase in the mean grain size is expected to promote the development of broader localized shear zones. This, in turn, may enhance fault relaxation but meanwhile increase the frictional strength of the fault.

5. Conclusions

In this study, we investigated the influence of initial grain size distribution on the frictional behavior of Dinantian carbonate simulated fault gouge using slide–hold–slide experiments. Our results demonstrated that the presence of fluids in carbonate gouges enhanced stress relaxation during hold periods, resulting in a lower coefficient of internal friction associated with creep shear stresses. In addition, fluid-saturated carbonate gouges exhibited higher absolute healing values and rates than their air-dry counterparts.

These behaviors were primarily governed by grain-scale cataclasis, which promoted the development of a fine-grained Principal Slip Zone (PSZ). The formation of this PSZ facilitated enhanced rates of pressure-solution creep. In contrast, peak and steady-state coefficients of internal friction were comparable under air dry and fluid-saturated conditions and were dominated by frictional sliding. Under wet conditions, the presence of a thin water film coating grain surfaces was sufficient to promote frictional sliding without significantly altering peak or steady-state friction.

We further proposed that frictional healing localized within the PSZ and was independent of the mean initial grain size of the starting material. In contrast, gouge relaxation occurring both within the PSZ and in the bulk gouge, was controlled by the thickness of the PSZ, and showed a positive correlation with the mean initial grain size. Our results indicated that the frictional strength of fluid-saturated carbonate faults was governed by the grain size within the PSZ. Consequently, grain-size-altering processes, such as dissolution of fine particles, will only affect fault strength if they operate within the PSZ. Finally, we employed CNS microphysical modeling to confirm that pressure-solution creep played a key role in controlling gouge deformation.

Conflict of Interest

The authors declare no conflicts of interest relevant to this study.

Availability Statement

The experimental data used in the study are available at the repository 4TU.ResearchData via Kane, Pluymakers, and Niemeijer (2025).

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