# Shear-enhanced electrical conductivity of synthetic quartz-graphite gouges: Implications for electromagnetic observations in carbonaceous shear zones

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14	Key Points:
15	• Experiments work shows that the presence of graphite can cause abrupt
16	increase in electrical conductivity at limited shear displacement
17	• Graphite–cortex clasts develop in the strain-localized zone
18	• Initiated slips at carbonaceous shear zones can be detected by monitoring
19	temporal electromagnetic anomalies
20	

#### 21 Abstract (248 words)

22 Graphite is considered as a material that promotes fault weakening and electrical 23 conductivity ( $\sigma$ ) enhancement at fault zones. We studied how shear deformation may affect the 24 evolution of friction and electrical conductivity of synthetic quartz (Qz)-graphite (Gr) mixtures 25 and, more importantly, whether the  $\sigma$  of the mixtures present visible changes at the beginning of 26 the simulated fault slip. Long-displacement friction experiments were performed on 1.2-2.3 mm-27 thick gouge specimens of varied Gr volume fraction ( $X_{Gr} = 0-100 \text{ vol.\%}$ ) under identical normal 28 stress (2 or 5 MPa), slip rate ( $\sim$ 1.0 mm/s), and N<sub>2</sub>-flushing conditions. The experimental results 29 suggested that the  $\sigma$  of the specimens with  $\geq$  4.6 vol.%  $X_{\rm Gr}$  abruptly increased under limited shear 30 displacement. With continued shear, the steady-state electrical conductivity ( $\sigma_{ss}$ ) increased by 31 more than seven orders of magnitude when  $X_{Gr} > 3.4$  vol.%, while the steady-state frictional 32 coefficient remained high (0.54–0.80) except for the specimens with  $X_{\rm G} > 13.6$  vol.%. The post-33 mortem microstructures revealed that the high  $\sigma_{ss}$  observed in the intermediate Gr content 34 specimens (3.4-13.6 vol.%) is associated with an *ad-hoc* fabric (graphite-cortex clasts) present 35 in the principal slip zone. For high Gr content, excess Gr flakes fill the pores and help develop 36 mechanically lubricated surfaces. We propose that low Gr content (i.e., as low as 3.4 vol.%) can 37 cause high conductivity anomalies in natural shear zones. Overall, the findings suggest that the 38 initiation of slips within carbonaceous shear zones can be detected by identifying unusual 39 temporal signals using electromagnetic stations.

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#### Plain Language Summary (172 words)

Crystalline graphite (Gr) can be enriched within fault zones due to mechanical or chemical 42 43 processes and is considered a material that promotes fault weakening and electrical conductivity 44 enhancement at fault zones. Geophysical observations suggest highly conductive anomalies in 45 the carbonaceous shear zones and low apparent resistivity anomalies prior to an earthquake. 46 Given this, we designed a novel experimental assembly to conduct electrical conductivity 47 measurements on Gr-bearing fault rocks along a fault-parallel direction during a progressive fault 48 slip in the laboratory. Our results revealed notably enhanced electrical conductivity under limited 49 shear displacement, corresponding to the beginning of the simulated fault slip. With continued 50 shear, the steady-state electrical conductivity increases by more than seven orders of magnitude as the Gr content exceeds 3.4 vol.%, while the steady-state frictional coefficient remains high until the Gr content exceeds 13.6 vol.%. Our results demonstrate that interconnected Gr networks are one of the main mechanisms that can explain high conductivity anomalies at shear zones and facilitate the detection of initiated slips in carbonaceous shear zones using electromagnetic stations.

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#### 57 **1. Introduction**

58 Foliated fault rocks with anastomosing-network fabric composed of weak minerals (mostly 59 phyllosilicate or carbonaceous materials [CMs]) are widely reported at natural fault zones (e.g., 60 Collettini et al., 2009; Collettini et al., 2019; Kuo et al., 2022; Manatschal, 1999; Oohashi et al., 61 2012). As revealed by their surface outcrops, CMs are present in several shear zones, especially 62 in deep ductile shear bands (e.g., Kedar et al., 2020; Lyu et al., 2020; Nakamura et al., 2015; 63 Puelles et al., 2014; Rawat & Sharma, 2011). Recent studies also report that CMs are 64 occasionally locally present in shallow brittle fault zones, such as in coseismic surface ruptures 65 (e.g., Kouketsu et al., 2017; Oohashi et al., 2012; Togo et al., 2011) or in drilling boreholes 66 across principal slip zones (e.g., Chen et al., 2016; Hirono et al., 2009; Kirilova et al., 2018b; 67 Kuo et al., 2014; Zulauf et al., 1999). The fraction of CMs varies among carbonaceous fault 68 zones. Most fault gouges contain ~1-36% CMs (Chen et al., 2017; Chen et al., 2016; Nakamura 69 et al., 2015; Wang et al., 2014). Some contain even 2-12 wt.% of crystalline graphite (Gr), a 70 special member of CMs (Manatschal, 1999; Oohashi et al., 2012). Due to diffusive mass transfer 71 or fluid precipitation during faulting, CMs can progressively become enriched toward the center 72 of fault zones (i.e., the principal slip zones) to accommodate large shear deformation (Kuo et al., 73 2014; Oohashi et al., 2012; Oohashi et al., 2013). Moreover, Gr-bearing shear zones show low 74 electrical resistivity in magnetotelluric (MT) surveys, generally in the range of 0.100–0.005 S/m 75 (e.g., Pous et al., 2004; Ritter et al., 2005; Wannamaker et al., 2002; Zhao et al., 2012).

Mechanical tests on binary (or ternary) mixtures of hard and weak minerals show that the shear strength of mixtures monotonically decreases with increasing content of weak minerals (Crawford et al., 2008; Moore & Lockner, 2011; Takahashi et al., 2007; Tembe et al., 2010). In particular, highly crystalline Gr has been characterized as a "dry" solid lubricant of fault zones because of its sheet structure held together solely by van der Waals forces (Kirilova et al., 2018a; Moore & Lockner, 2004). Previous studies have shown that the steady-state frictional coefficient of Gr can remain at very low values (between ~0.1 and ~0.2) over a wide range of slip rates  $(5 \times 10^{-4} - 1.3 \text{ m/s})$  (Kirilova et al., 2018a; Oohashi et al., 2011; 2013). Even a small amount of Gr (~10 vol.%) in a fault gouge can mechanically smear on the principal shear plane. It makes fault rocks dramatically weaker than expected when considering Byerlee's law (0.6–0.85 in friction coefficient, Byerlee, 1978; Oohashi et al., 2013; Rutter et al., 2013).

87 Grain-boundary Gr films (a few ppm), with thicknesses ranging from a few to several tens 88 of nanometers, can produce highly interconnected conductive networks. Their electrical 89 conductivity reaches 0.005-0.010 S/m (Duba & Shankland, 1982; Frost et al., 1989; Glover & 90 Vine, 1992; Mareschal et al., 1992). Such films may exist in the range of crustal depth (Selway, 91 2013). They have been considered as a potential mechanism explaining high conductivity 92 anomalies in crustal shear zones (Chen et al., 2017; Glover & Ádám, 2008; Haak et al., 1997; 93 Monteiro Santos et al., 2002). Moreover, apparent resistivity anomalies prior to an earthquake 94 (typically descending by 1~7%) detected by electromagnetic stations have been considered as a 95 precursor factor in medium- or short-term earthquake prediction (e.g., Du, 2011; Honkura et al., 96 2013; Lu et al., 2016; Madden et al., 1993; Zhao & Qian, 1994) despite their strong spatial 97 anisotropy. Nover et al. (2005) showed that shearing deformation can enhance the electrical 98 conductivity of carbon-bearing rocks by about three orders of magnitude. Glover & Ádám (2008) 99 attributed this enhancement to the smearing effect and proposed that this effect can explain many 100 precursory and coseismic geoelectric phenomena observed in nature (Mathez et al., 2008; 101 Roberts et al., 1999). However, to date, there are insufficient systematic real-time observations of 102 the mechanical and electrical characteristics of shearing carbon-bearing gouges. It leads to 103 incomplete theoretical support for explaining the origin of high conductivity anomalies in deep 104 shear zones and effectively obtaining the frictional slip information in shallow fault zones.

In this study, we used a rotary shear apparatus to conduct continuous electrical conductivity measurements on controlled-dry, synthetic, Gr-bearing gouges along the fault-parallel direction during progressive fault slip. The experiments were conducted at fixed velocity ( $\sim$ 1 mm/s), normal stress (2 or 5 MPa), and under ambient temperature, N<sub>2</sub> atmosphere conditions. Results showed that an increasing Gr content can effectively reduce the frictional strength, while an initial very limited shear displacement causes an abrupt enhancement of the electrical conductivity. We further investigated the microstructure evolution of specimens with different Gr contents and found that the high electrical conductivity observed might be related to the development of Gr flakes on the grain boundaries. Our work gives insights into the coupling effect between frictional strength and electrical conductivity of Gr-bearing fault zones. Additionally, it offers experimental evidence for detecting initiated slips using electromagnetic approaches.

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# 118 **2. Materials and methods**

119 2.1. Starting materials

120 The starting materials used in our experiments were commercial Gr powders (Xilong 121 Scientific Co., Ltd., analytical grade, > 98.5% purity, Figure S1a in Supporting Information) and 122 Quartz (Qz) particles (collected from Fengyang County, Anhui Province, China P.R., > 99.3% purity, Figure S1b in Supporting Information). The sizes of the Qz particles were determined by 123 124 laser diffraction analysis (Microtrac S3500), which resulted in a median diameter of 12.2 µm and 125 a size distribution comparable to that of natural fault gouges (Chen et al., 2017) (Figure S1c in 126 Supporting Information). Synthetic fault gouges were prepared by mixing the Qz particles with Gr powders in contents of 0, 3, 4, 5, 6, 9, 10, 12, 15, 25, 50, and 100 wt.%. According to the 127 particle densities (2.31 g/cm<sup>3</sup> for Gr and 2.66 g/cm<sup>3</sup> for Qz) measured by the true density 128 129 analyzer (AccuPyc II 1340, errors  $\pm 0.03\%$ ), the estimated volumetric percentages of Gr in the 130 mixtures were 0.0, 3.4, 4.6, 5.7, 6.8, 10.2, 11.3, 13.6, 16.9, 27.7, 53.5 and 100 vol.%, respectively. 131

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## 2.2 Experimental assembly adapted to friction-conductivity measurements

The experiments were conducted using the low- to high-velocity rotary shear apparatus installed at the Institute of Geology, China Earthquake Administration (IGCEA) (Figures 1a–1c). For this instrument, the variation of axial force (i.e., normal stress) could be controlled within 2– 3%, the resolution of shear displacement is ~30  $\mu$ m, and the accuracy of the measured shear torque is greater than 99% (Ma et al., 2014). We adapted a ring-shear setup for gouge-type friction experiments to monitor the transient electrical conductivity (real-time response of the electrical conductivity,  $\sigma$ ) of simulated faults (Figure 1d) in their fault-parallel direction (Figure 140 1e). For testing the assembly, a simulated gouge layer with  $\sim 2.0$  mm thickness was uniformly 141 placed between a pair of 40 mm-long corundum hollow cylinders with an inner diameter  $(l_i)$  of 142 28 mm and an outer diameter ( $l_0$ ) of 40 mm, respectively. In previous experiments that used the 143 ring-shaped assembly, before our adaptation, the gouge layer was typically confined by the tightly fitted outer and inner Teflon<sup>®</sup> sleeve/cylinder to minimize gouge extrusion (e.g., Boulton 144 et al., 2017; Hou et al., 2012; Yao et al., 2013a; Yao et al., 2013b). However, in the designed 145 146 setup, to allow the electrical conductivity measurement, two titanium-alloy electrodes (a loop and a centered vertical cylinder) were embedded into the outer Teflon<sup>®</sup> sleeve and inner cylinder, 147 respectively (Chen, 2022; Han et al., 2019). To avoid direct contact between the upper (rotary) 148 149 corundum cylinder and the electrodes, the gap between them was kept at  $\sim 100 \ \mu m$ . We note that 150 a small quantity of gouge was expected to extrude into the gaps during the experiments. It could 151 presumably cause some uncertainty in the friction data. One or two lead wires led respectively 152 from the two stainless steel screws on the two electrodes (Figure 1b). They were connected to the 153 Keithley instruments (Tektronix Company, U.S.) used to measure the electrical resistance ( $R_E$ ). 154 In addition, a suit of plastomers fixed the three-layered outer sleeve to the lower corundum 155 cylinder. Finally, to achieve a dry and anoxic environment, the whole assembly was enclosed by 156 a transparent polymethylmethacrylate (PMMA) vessel. A (high purity) N2 atmosphere was 157 maintained inside the vessel during the experiment.

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#### 159 2.3 Experimental procedure and data processing

A total of 19 experiments were performed on the different Gr–Qz mixtures under constant normal stresses ( $P_n = 2 \text{ or } 5 \text{ MPa}$ ) and room temperature conditions. The specimens were ovendried at  $\geq 75 \text{ °C}$  for > 24 h prior to the experiments. After setting up each specimen assembly, it was first compacted at the target  $P_n$  for 2–3 h and then sheared at a constant slip rate of 0.83– 1.00 mm/s under dry conditions.

During the experiments, three Keithley instruments (6514 System Electrometer, 2182A Nanovoltmeter, and 6221 DC and AC Current Source) were used to measure  $R_{\rm E}$ . These instruments are commercial products. They enable fast, precise, high-sensitivity measurements of various electrical parameters and have been widely used for constraining the electrical properties 169 of geological materials (e.g., Hou et al., 2021; Yamashita et al., 2014; Zhuang et al., 2021). As 170 the  $R_{\rm E}$  values of our simulated specimens varied by almost 14 orders of magnitude (from 100 G $\Omega$ 171 to 1 m $\Omega$ ), we used three different configurations and measurement modes depending on the  $R_{\rm E}$ range. Thereby, the electrical potential (E) and direct current (I)  $[R_E = E/I, 4 \text{ wires setup}]$  or  $R_E$  (2) 172 173 wires setup) of the specimens were acquired. Details on the resistance measurement models and 174 corresponding measurement accuracies are presented in Table 1. The  $R_E$  values were obtained by 175 subtracting the background levels (0.0041  $\Omega$ ). They contained the electrical resistance of the 176 aluminum wires in the assembly and the Keithley instruments, which were assessed from the 177 electrode-to-electrode measurement. The  $\sigma$  values were calculated by taking the inverse of the  $R_{\rm E}$ 178 values and normalizing them with the scale as follows:

$$\sigma = \frac{\ln(l_o/l_i)}{2\pi\delta R_E} \tag{1}$$

179 where  $\delta$  is the thickness of the simulated gouge layer (mm). In some experiments, we paused the 180 motor for ~5 min to switch the measurement mode due to technical issues or unexpected changes 181 in  $\sigma$ . The consistency of the  $\sigma$  results between two modes demonstrates the relative accuracy of 182 our measurements.

Besides recording electrical data, the axial load, axial displacement, torque, and upper piston rotation were also recorded at 20 Hz using a digital data recorder (KYOWA EDX-100A). The raw data were processed to obtain  $P_n$ ,  $\delta$ , equivalent slip velocity ( $v_e$ , m/s), equivalent shear stress ( $\tau$ , MPa), and apparent friction coefficient ( $\mu = \tau/\sigma_n$ ) vs shear displacement (D) (Ma et al., 2014). The  $\mu$  value was calculated after correcting the Teflon<sup>®</sup> friction (Hou et al., 2012). As  $R_E$ was also recorded at 20 Hz using the Keithley instruments, we carefully synchronized the two recording systems by matching the feature points of  $R_E$ .

For quantitative comparison between the two quantities, we determined several critical parameters from the  $\mu$  and  $\sigma$  vs D curves (Table 2, Figures 2–3). For instance,  $\mu_{ss}$  and  $\sigma_{ss}$  are the nominal (quasi-) steady-state frictional coefficient and electrical conductivity achieved at long displacement. They were obtained from the arithmetically average value of  $\mu$  in steady state and the logarithmically average value of  $\sigma$  in steady state, respectively.  $D_{\mu ss}$  and  $D_{\sigma ss}$  are the corresponding characteristic displacements. Moreover, we defined  $D_{\sigma ch}$  as the critical slip displacement for abrupt electrical conductivity enhancement (i.e.,  $\sigma$ -jump phenomenon). Namely, 197 the  $\sigma$  curve, for the first time, shows an increase by more than one order of magnitude from the 198 low initial level ( $\sigma_0 < 0.01$  S/m), or more than 1.5 times from a high initial level ( $\sigma_0 > 0.01$  S/m). 199 For details on the data processing and determination of the critical parameters, we refer to Text 200 S1 and Figure S2 in Supporting Information. 201 After the experiments, a scanning electron microscope (SEM, Zeiss Sigma-0380) was used

to examine the microstructural developments as a function of the Gr content (Figure 1e). The scanning electron microscope was operated at an acceleration voltage of 15 kV in both the backscatter electron (BSE) and secondary electron (SE) modes. Microstructural images were taken from the corresponding Au-coated thin sections.

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#### 207 **3. Results**

The experimental data obtained from the 19 experiments (with the synthetic fault gouges) are presented in Table 2. As described in section 2.3, two series of data were obtained using our rotary-shear friction apparatus adapted for conducting transient electrical conductivity measurements.

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#### 213 3.1 General mechanical and electrical behaviors

214 Figure 2 presents the mechanical (panels a–b) and electrical (panels c–d) behaviors of the 215 Gr-Qz mixture specimens. They were sheared at a normal stress of 2 MPa as a function of slip 216 displacement in both logarithmic (panels a and c) and linear (panels b and d) scales. With low to 217 intermediate Gr contents (< 25 wt.%), the specimens exhibited peak friction coefficients of 0.49– 218 0.67 at less than 0.17 m slip displacement (Figure 2a). Then, it was followed by slip 219 strengthening to  $\sim 1$  m and overall high friction levels (0.54–0.80) in the end (Figure 2b). With 220 high Gr contents (25–50 wt.%), the peak friction reached 0.40-0.51 at a displacement of ~1 mm, 221 followed by dramatic slip weakening with steady-state friction coefficients of 0.10–0.19. At a 222 high normal stress of 5 MPa, similar frictional behaviors were evident for both low and high Gr 223 contents (Figure 3a).

The specimens showed large variations in electrical conductivity of up to 14 orders of magnitude (i.e., from  $10^{-11}$  to  $10^3$  S/m), depending on the Gr content and shear displacement. (1) For Gr content < 4 wt.%,  $\sigma$  decreased slightly with initial shear displacement. (2) For Gr content of 4–10 wt.%, the specimens showed intermediate  $\sigma_{ss}$  values in the range of 10<sup>-3</sup>–0.3 S/m. Interestingly,  $\sigma$  increased remarkably with the slip progression after  $D_{\sigma ch}$  by more than six orders of magnitude. (3) Specimens with Gr contents > 12 wt.% showed slight increases (a few times) in  $\sigma$  with slip. The  $\sigma$  maintained high values (> 0.1 S/m) throughout the experiments.

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# 232 3.2 Comparison of characteristic displacements

233 The results of the  $\sigma$ -D and  $\mu$ -D data by Gr content are presented in Figure 4. To reveal the 234 possible links between  $\sigma$  and  $\mu$ , we highlight the  $\sigma$  and  $\mu$  evolution at displacements between  $D_{\sigma ch}$ 235 and  $D_{\sigma ss}$ , during which  $\sigma$  increased significantly.

236 The initial nearly-linear portions of the  $\mu$ -D curves reflect the elastic shear loading 237 processes. Because the displacements in all  $\mu$ -D and  $\sigma$ -D plots are precisely the load point 238 displacements that embody the shear deformation of the entire testing system. The displacements 239 at which the  $\mu$ -D curves deviate from straight lines are the starting points of the shear 240 deformation in the gouge layers (see the circle symbols in Figures 4d–4f, hereafter referred to as 241  $D_0$ ). Thus, the difference between  $D_0$  and  $D_{ach}$  represents the shear displacement required to 242 initiate the generation of a continuous electrically conductive layer associated with the shear 243 deformation of the Gr–Qz mixtures. Since the  $D_0$  values of all specimens are small and not very 244 different from one another (typically  $\sim 0.15-0.28$  mm), we plotted  $D_{\rm orch}$  against the Gr content in 245 Figure 4g and Figure 4h, while  $D_{\mu ss}$  and  $D_{\sigma ss}$  were plotted for comparison. As the Gr content 246 increased, the  $D_{\sigma ch}$  value decreased from 0.5 m, via 4 mm, to 0.2 mm; similarly,  $D_{\sigma ss}$  decreased 247 from 1.2 m, via 100 mm, to 3.0 mm. In contrast,  $D_{\mu ss}$  seemed to be independent of the Gr content, thereby remaining at high values (mostly between 0.3 and 0.6 m). Therefore,  $D_{\sigma ch}$  and  $D_{\sigma ss}$  were 248 249 significantly lower than  $D_{\mu ss}$  at Gr content higher than ~9 wt.%. Consequently, for the mixtures with 4–12 wt.% Gr, the critical slip displacement for the  $\sigma$  jump ( $D_{ach}$ ) was limited (as low as 0.2 250 251 mm) and smaller than the displacement for reaching the frictional steady state ( $D_{\mu ss}$ ) (see the start 252 of the bold curves in Figures 4d–4e). The  $\sigma$  jump occurred before the peak friction, even in the case of mixtures with high Gr content (15–50 wt.%) (see the start of the bold curves in Figure 4f). 253

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#### 3.3 Microstructural evolution in steady state

As revealed by the BSE images of the epoxied post-mortem specimens, with increasing Gr content (0, 3, 9, and 25 wt.%), the specimens became increasingly cohesive after the experiment. The pure Qz gouge was the most fragile, showing a loose structure, whereas the high Gr content gouge layer could be recovered as an entire piece (Figure 5). A slickenside surface ornamenting the discrete shear surface of a high-Gr portion specimen was observed after the experiment (attached picture on top of Figure 5d), while a dark surface appeared on the surface of a low-Gr portion specimen (attached picture on top of Figure 5c).

263 The pure Qz gouge did not show a discernable strain localization zone (SLZ). However, 264 discrete inclined openings were visible over the entire thickness, mostly along the Riedel shear 265 (R shear, Logan et al., 1992), oriented at a low angle of  $10^{\circ}$ -30° to the shear direction (Figure 266 5a). In contrast, remarkable shear bands were developed at the upper rotary boundaries in the 267 other mixture specimens, especially those with low Gr contents (3 and 9 wt.%, Figures 5b and 268 5c). In these two specimens, the angular fractured Qz particles of the lowest layer had relatively 269 large grain sizes, similar to that of the pure Qz gouge (Layer III). The localized band was further 270 divided into two layers (see the yellow dotted lines for the boundaries, Figures 5b and 5c). 271 Generally, the middle layer was homogeneous, and its average grain size ( $\sim 5 \mu m$ ) was lower than 272 that of the least deformed layer. The uppermost layer had the finest grain size, mostly lower than 273  $\sim 1 \mu m$ . In a certain portion of this layer, agglomerated clumps of extremely fine Qz particles 274 developed, characterized by thicknesses of  $\sim 10 \ \mu m$  and lengths varying from a few tens to a few 275 hundreds of micrometers. Locally, they were cut by the R shear offset, manifested as discrete 276 stripes parallel to the shear direction (Figures 5b and 5e). Such discrete clumps were interpreted 277 as the migration of the microslip zone during extreme shearing deformation, in accordance with 278 previous reports (Yao et al., 2013a; Yao et al., 2013b). The enlarged pictures indicate that these 279 clumps were characterized by homogenous Oz grain size distribution and had extremely low 280 porosity (< 5%, estimated based on image analysis, Figure 5e), while the remainder of the upper 281 layer had a broader grain size distribution (varying from 0.1 to 1.0 µm) and relatively high 282 porosity (20–30%, Figure 5f). Gr flakes, with widths of up to several tens of nanometers, filled the space between the Qz particles to form anastomosing networks (Figure 5f). Consequently, the 283

microslip zone was developed from the middle layer and grew toward the upper boundary as the slip proceeded, as revealed by the comparison of the microstructures between Layers I and III. The microstructure of the high Gr specimen (25 wt.%) showed extremely localized deformation, and the shining surface on the specimen formed a 10–20 µm layer (Figure 5d). The enlarged picture shows deflected Gr flakes that formed anastomosing networks around broken Qz particles.

290 The Gr and the epoxy resin were difficult to distinguish at the submicron scale based on 291 SEM images. Thus, we directly observed the unepoxied specimens of 12 wt.% Gr-bearing 292 recovery specimen (LHV2429) under a top view perspective (Figure 6a). The upper boundary 293 showed a relatively smoothed surface, consisting of fine Qz particles of  $\sim 1-2 \mu m$  with sub-294 angular shape and submicron Gr flakes. It was consistent with Layer I identified in the thin 295 section (Figure 6b). The enlarged image shows that the individual Qz particles were pasted on 296 the surface by a large amount of small Gr flakes (Figure 6c). In contrast, the broken surface 297 beneath the upper surface (see its position in Figure 6a) had relatively large particles (~10 µm) 298 (Figure 6d). The individual Qz particles were mostly angular without visible foliation, while Gr 299 appeared as isolated grains, suggesting relatively small deformation (Figure 6e). Note that 300 numerous small particles were seen to be attached to the surfaces of Qz particles, but most were 301 Qz debris (Figure 6f). All these features were similar to those observed in Layer III of specimens 302 with intermediate Gr wt.% (i.e., 3 and 9 wt.%).

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#### 304 **4. Discussion**

305 4.1 Mechanical and electrical behaviors of graphite-bearing faults

4.1.1 Steady-state friction and conductivity relations of varying  $X_{Gr}$  specimens

To facilitate the comparison with previous works, the Gr fractions of all specimens were transformed from weight percentages to volume percentages ( $X_{Gr}$ ). Figure 7 shows  $\mu_{ss}$  and  $\sigma_{ss}$ plotted against  $X_{Gr}$  for all specimens.

The  $\mu_{ss}$ - $X_{Gr}$  data determined a threshold value (~13.6 vol.%), i.e., the  $X_{Gr}$  at which the frictional strength starts to decrease. A systematic increase of  $X_{Gr}$  from ~13.6 to 30 vol.% led to a nonlinear  $\mu_{ss}$  decrease from 0.72 to 0.14. Thereafter, when  $X_{Gr}$  changed from 30 to 100 vol.%,  $\mu_{ss}$  gently approached  $\mu_{Gr}$  (~0.1). Oohashi et al. (2013) conducted frictional experiments on Gr–Qz mixtures under similar conditions ( $\sigma_n = ~2$  MPa, dry,  $v_e = 0.2-56.0$  mm/s,  $X_{Gr} = 0-100$  vol.%) and observed similar weakening trends as  $X_{Gr}$  increased (although they did not measure  $\sigma_{ss}$ ). Following Oohashi et al. (2013), the relationship between  $\mu_{ss}$ – $X_{Gr}$ , in this study, can be described by

$$\mu_{\rm ss} (X_{\rm Gr}) = \frac{\mu_{\rm Qz} - \mu_{\rm Gr}}{1 + (X_{\rm Gr} / X_{\rm cw})^S} + \mu_{\rm Gr},$$
(2)

where  $\mu_{Qz}$  and  $\mu_{Gr}$  are the frictional coefficients of pure Qz and pure Gr, respectively. The  $\mu_{Qz}$ (0.73) was taken as the mean value of  $\mu_{ss}$  for the low- $X_{Gr}$  specimens (< 13.6 vol.%), and  $\mu_{Gr}$  was taken as 0.10 based on the  $\mu_{ss}$  values of pure Gr flakes.  $X_{cw}$  is the critical  $X_{Gr}$ , at which friction reduces to the averages of  $\mu_{Qz}$  and  $\mu_{Gr}$ . The power exponent *S* indicates the slope of the weakening with increasing  $X_{Gr}$ . The fitted parameters are presented in Table 3, and the correlation coefficient ( $R^2$ ) is 0.94.

324 The  $\sigma_{ss}$  results show a significant sharp increase when  $X_{Gr}$  exceeds 3.4 vol.% (i.e., by more than 7 orders of magnitude from 3.4 to 4.6 vol.%, Figure 7) and the  $\sigma_{ss}$  of pure Gr flakes (10<sup>3</sup> 325 S/m) is much greater than that of pure Qz particles ( $10^{-11}$  S/m). The enhancement of  $\sigma$  caused by 326 327 Gr has been observed in high pressure and/or high temperature experiments under static 328 conditions (Chen et al., 2017; Wang et al., 2013). In particular, Chen et al. (2017) explored the 329 electrical conductivity of similar Qz-Gr mixtures under non-sheared deformation and a wide 330 range of stress from 0.1 to 300.0 MPa. Following a previous study, percolation theory, i.e., a 331 model describing well the current transport properties through porous mediums (Stauffer & 332 Aharony, 2003), can explain the trend of the  $\sigma_{ss}$ -X<sub>Gr</sub> curve via the following equation (Gueguen 333 & Dienes, 1989):

$$\sigma_{\rm ss} (X_{\rm Gr}) = \sigma_{\rm Qz} + \left(\sigma_{\rm Gr} - \sigma_{\rm Qz}\right) \left[\frac{\alpha(X_{\rm Gr} - X_{\rm c})}{1 - \alpha X_{\rm c}}\right]^r, \qquad (X_{\rm Gr} \ge 3.4\%) (3)$$

where  $\sigma_{Qz}$  and  $\sigma_{Gr}$  are the electrical conductivities of the relatively insulated matrix (Qz) and conductive inclusion (Gr),  $\alpha$  is the geometric factor of the conductor (Gr), and *r* is a nondimensional parameter.  $X_c$  is the threshold value of  $X_{Gr}$ . It represents the critical volume fraction of the conductor (Gr flakes in this study) for forming interconnected networks in the specimens and depends on the geometry of the Gr flakes (Stauffer & Aharony, 2003). We note that this study focuses on the effect of shear, especially the experimental data of low to

- intermediate  $X_{Gr}$  specimens (< 20 vol.%). Therefore, data of high- $X_{Gr}$  specimens (> 20 vol.%) are more scarce and cannot provide a better fitting for the power law growth. The fitted parameters of Equation 3 are presented in Table 4, and the correlation coefficient ( $R^2$ ) is 0.98. Other fitting details of the  $\mu_{ss}$ - $X_{Gr}$  and  $\sigma_{ss}$ - $X_{Gr}$  relationships can be found in Text S2.
- 344

345 4.1.2 Representative microstructure interpretation

346 The critical  $X_{\rm Gr}$  value for a  $\mu_{\rm ss}$  decrease (~13.6 vol.%) was greater than that for a  $\sigma_{\rm ss}$  increase 347 (3.4 vol.%) (as shown by the comparison between the  $\mu_{ss}$ - $X_{Gr}$  and  $\sigma_{ss}$ - $X_{Gr}$  curves in Figure 7). It 348 may be caused by the specific microstructural variations of the Qz-Gr mixtures. Consequently, we divided this system into three regimes bounded by 3.4 and ~13.6 vol.%  $X_{Gr}$ , respectively. For 349 350 carbon-bearing rocks, the efficient electrical conductivity enhancement is mainly derived from 351 the microstructure of interconnected grain-boundary CMs (Duba & Shankland, 1982; Frost et al., 352 1989; Mareschal et al., 1992). Associated with the SEM images in Figures 5 and 6, we propose 353 four classes of microstructures dependent on  $X_{Gr}$  (T1–T4, see the schematic diagrams beneath the 354 coordinate system of Figure 7) as follows:

355 (a) Pure Qz gouge ( $X_{Gr} = 0$ ). It exhibited insulating (~10<sup>-11</sup> S/m) and high-shear strength 356 properties ( $\mu_{ss} \approx 0.73$ ). A fault slip formed at the contact between Qz particles, and R-shear 357 surfaces developed throughout the entire mixture.

(b) Low  $X_{\text{Gr}}$  regime (0% <  $X_{\text{Gr}} \le 3.4\%$ ). Similar to pure Qz gouge, it presented low electrical 358 conduction (~10<sup>-10</sup> S/m) and high-level frictional strength (0.79-0.85). Three layers with 359 360 different Qz grain sizes (Layers I-III) developed, and similar results have been reported in 361 natural gouges (Hou et al., 2012; Wang et al., 2014). The non-foliated layer (Layer III), in the 362 bottom, comprised isolated angular Qz particles mixed with Gr flakes. The upper ~400 µm SLZ 363 consisted of an ~200 µm Layer I, and an ~200 µm Layer II was produced near the side of the specimen–cylinder boundary (grey layers in Figure 7). Moreover, low  $\sigma_{ss}$  of the mixtures in this 364 365 regime suggests the conductive Gr flakes cannot interconnect in this microstructure.

366 (c) Intermediate  $X_{Gr}$  regime (3.4% <  $X_{Gr \leq}$  13.6%). It was characterized by  $\sigma_{ss}$  enhancement 367 with increasing  $X_{Gr}$  (10<sup>-3</sup> S/m <  $\sigma_{ss}$  < 4 S/m). It also presented a three-layered microstructure 368 (Layers I–III) that differed from that of the low  $X_{Gr}$  regime. The Gr flakes were more frequent in 369 the pore spaces supported by the Qz framework with increasing  $X_{Gr}$ . Unimpregnated 370 microstructures of the slip surfaces in this regime showed a large number of tiny Gr flakes (~10 371 nm) pasted on the surface of Qz particles (Figures 6b and 6c). Thus, we propose that the SLZ of 372 Qz-Gr mixtures developed the fabric of graphite-cortex clasts (GCCs), i.e., comminuted 373 subangular Qz clasts (< 1  $\mu$ m) were surrounded by a cortex of concentric Gr layers (~10 nm), 374 which were composed of ultrafine pulverized Gr flakes (see the schematic cartoon extended from 375 the T3 texture). In contrast, Gr flakes in this regime could not be enriched to form lubricated slip 376 surfaces due to limited Gr fraction, and, therefore, the friction remained high ( $\mu_{ss} = 0.54-0.84$ ). 377 Moreover, GCCs may also exist in the low  $X_{Gr}$  regime, but due to the low  $X_{Gr}$ , they may not be 378 electrically connected.

379 (d) High  $X_{Gr}$  regime (13.6%  $\leq X_{Gr} < 100\%$ ). It exhibited high  $\sigma_{ss}$  values (> 1 S/m) and low 380  $\mu_{ss}$  values (mostly < 0.2). In particular, specimens in this regime had an initially conductive 381 structure ( $D_{\sigma ch} < 0.2$  mm). Frictional weakening occurred during the experiment, and the 382 required slip distances were uniform ( $D_{\mu ss} = 0.2-0.3$  m, Figures 4g-4h). When the  $X_{Gr}$  was 383 enhanced, the frictional level approached a pure Gr powder ( $\mu \approx 0.1$ ). In this case, the specimen 384 usually presented a narrow boundary shear band (Figure 5d) instead of a dispersive shear zone (cf. low  $X_{Gr}$  regimes). The single shear zone (or shining surface) with an ~20 µm thickness was 385 386 similar to that of the abandoned shear surface (Qz clumps in Figure 5e).

387

#### 388 4.1.3 Mechanisms responsible for electrical conduction and slip weakening

As addressed earlier, the  $\sigma$  of pure quartz and Gr–Qz mixtures with  $X_{\text{Gr}} \leq 3.4\%$  decreased with initial shear displacement by several orders of magnitude. We interpreted that the initial current flow was mainly through the grain contacts of large grains that developed under initial static compaction, and that the transient decrease of electrical conductivity was due to the destruction of large contacting asperities by initiating slip. A similar process has been proposed by Yamashita et al. (2014) to explain the changes in electrical conductivity of gabbro specimens sheared at subseismic velocities.

396 For the Gr–Qz mixtures in the intermediate  $X_{\text{Gr}}$  regime (3.4% <  $X_{\text{Gr}} \leq 13.6\%$ ),  $\sigma$  showed 397 unstable fluctuations and eventually increased by more than six orders of magnitude during 398 progressive slip from  $D_{\sigma ch}$  to  $D_{\sigma ss}$ . In the steady state, they exhibited highly frictional strength 399 (after  $D_{\mu ss}$ ) and electrical conduction (after  $D_{\sigma ss}$ ). Meanwhile, the SEM images showed a three-400 layered microstructure. We interpret that the conductive pathways mainly occurred in Layer I in 401 the form of GCC fabric for the following reasons:

402 Layer III was almost undeformed and resembled an original preslip zone of the mixture. 403 The fractures in the Qz particles may result from the axial compaction derived from the normal 404 stress on the specimen. During the progressive slip, the original Qz particles were comminuted to 405 micron- and even submicron-sized clasts, while subsequently producing an  $\sim 200 \ \mu m$  intensively 406 foliated layer (Layer I) and an ~200 µm weakly foliated layer (Layer II), respectively. Compared 407 with Layer I, Layer II exhibited a relatively larger Qz clast size and weak strain localization lying 408 on the transitional phase between Layers I and III. Therefore, from the bottom to the top of the 409 bulk gouge, the shear strain gradually increased, and the original Qz particles experienced a 410 series of processes. They were axial compression, fragmentation, attrition, comminution, shear-411 induced clumping, and finally, attracting the ground Gr flakes to generate the GCC fabric in the 412 SLZ. Several Qz clumps consisting of aggregated ultrafine Qz clasts with a thickness of  $\sim 10 \ \mu m$ 413 were abandoned in Layer I. The pore-filled Gr flakes between the Qz particles also underwent 414 microstructural evolution from disconnection to interconnection. The instability of  $\sigma$  from  $\sigma_{ch}$  to 415  $\sigma_{ss}$  corresponds to this process of microstructural transformation.

416 Our proposed GCC fabric is an unusual spherical aggregate. To our knowledge, this 417 microstructure has not been published in other studies, including those of unsheared Gr-Qz 418 mixtures (Chen et al., 2017) and sheared Gr-Qz mixtures at various slip rates (Oohashi et al., 419 2013). Its overall appearance resembles those of clay-clast aggregate or clast-cortex aggregate 420 (CCA) fabrics. They were reported in previous investigations of shallow-depth seismogenic 421 faults in the field or in rotary-shear experiments in laboratory (Boullier et al., 2009; Han & 422 Hirose, 2012; Kim et al., 2022; Rempe et al., 2014; Sawai et al., 2012; Ujiie & Tsutsumi, 2010). 423 We hypothesize that the formation process of the high friction-conduction GCC fabric is similar 424 to that of the CCA formation model proposed by Boutareaud et al. (2010), i.e., electrostatically 425 charged Qz particles by fractoemission and triboelectric effect attract Gr flakes to the "negative" 426 Oz surface (Huang, 2002; Yoshida et al., 1997). Moreover, our results demonstrated that the 427 GCC fabric was developed in samples with relatively low Gr contacts and required a relatively 428 large shear displacement under room-dry conditions. These conditions are also similar to the
429 CCA formation conditions suggested by Han & Hirose (2012); Kim et al. (2022); Rempe et al.
430 (2014).

431 The Gr–Qz mixtures in the high  $X_{\rm Gr}$  regime (13.6%  $\leq X_{\rm Gr} < 100\%$ ) showed high  $\sigma$  values 432 before shear deformation (> 0.3 S/m), suggesting that the Gr flakes were texturally 433 interconnected upon initial compaction. With continued shear,  $\sigma$  further increased to even higher 434 values (> 1 S/m), which can be attributed to the formation of the shear band. As reflected by the 435 microstructure, the reduced porosity of the SLZ caused an apparent enrichment of Gr flakes and 436  $\sigma$  enhancement (Figure 5d). It was supported by the previous compaction experiments that 437 elevating the static stress could cause reduced porosity and, thus, the increase in electrical 438 conductivity (Chen et al., 2017). Meanwhile, specimens in this high  $X_{\rm Gr}$  regime were expected to 439 readily weaken when subjected to shear deformation (Oohashi et al., 2013). We infer this is 440 because of the apparent enrichment of Gr flakes due to porosity reduction and the development 441 of slip-lubricated surfaces (Figure 5d). Interestingly, the thicknesses of the slip surfaces (10-20  $\mu$ m) were comparable to that of the Qz clumps developed in the intermediate  $X_{Gr}$  samples (see 442 443 Figure 5e vs. Figure 5d). As indicated earlier, the latter were abandoned microslip zones during 444 progressive slip, reflecting the migration of localized deformation. We infer that the single slip 445 surface seen in the high  $X_{\rm Gr}$  regime derived from a microslip zone developed at the earlier stage 446 of shearing, whose resistance to shear is expected to be lower than that of the bulk layer. In all, 447 these results suggest a strong interplay between the mineralogy, structure, and mechanical 448 behavior of a fault.

449 Finally, we have used the percolation model (Equation 2) to fit the relationship between  $\sigma_{ss}$ -450  $X_{\rm Gr}$  of the Gr–Qz mixtures with > 3.4%  $X_{\rm Gr}$ . The applications of the percolation theory 451 concerning the electrical current transport properties assume that the conductor is randomly 452 located in a stable and isotropic structure (Stauffer & Aharony, 2003). Although we applied the 453 percolation model to describe the conductive transport properties of a sheared fault gouge, the 454 corresponding microstructure is assumed to have reached a similar quasi-steady state, reflecting 455 an average structure of millions of particles. Admittedly, the proposed conductive textures in the 456 intermediate to high  $X_{Gr}$  regimes in Figure 7 are anisotropic. However, the theory model can well express the relationship between  $\sigma_{ss}$ -X<sub>Gr</sub> under shear deformation in this study. It suggests that 457

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the percolation model could have a much wider application. Nevertheless, it still requires furtheradaption of the percolation model to incorporate the anisotropic transport structure.

460 One possible development would involve the dimensions in which the conductive material 461 (i.e., Gr) is distributed. Nominally, one could consider that the GCCs (graphite-cortex clasts) 462 fabric is a two-dimensional (2D) structure, i.e., the Qz particles in the shear active zone are fully 463 covered by Gr flakes (the schematic cartoon in Figure 7). However, as suggested by a simple 464 calculation assuming Gr flakes of varied thicknesses (10-50 nm), obtaining such a structure 465 requires a Gr content of at least 5.7 vol.%, higher than the threshold value of 3.4 vol.% obtained 466 in the present experiments. At this point, an electrically conductive structure in one dimension 467 (1D), i.e., the Gr flakes are attached end-to-end on the Qz surface to reach the electrodes, can 468 help settle the discrepancy. The real GCC fabric in the samples might fall between 1D and 2D. 469 Moreover, this does not conflict with the observation that much higher Gr contents (>13.6 vol%) 470 are required to cause significant frictional weakening at otherwise the same conditions (Figure 7). 471 This is because the frictional resistance of a shearing gouge is collectively determined by all the 472 grain contacts within the active shear zone, such that the weakening would be more favorable 473 when the Gr flakes somehow form a 2-D structure, requiring a higher Gr content. Nonetheless, at 474 present, it is difficult to justify the aforementioned models. A combination of numerical 475 simulations based on more sophisticated microstructure characteristics and updated percolation 476 models is warranted in the future.

477

#### 478 4.1.4 Implications for high conductivity anomalies

The high conductivity anomalies in the fault zones observed by MT surveys are generally limited within the range of 0.100–0.005 S/m (e.g., Pous et al., 2004; Ritter et al., 2005; Wannamaker et al., 2002; Zhao et al., 2012). As illustrated in Figure 8, our data suggest that the Qz–Gr mixtures containing 5.4–8.1 vol.% Gr explain such observations (yellow stars in Figure 8). However, this range is higher than those reported at fault zones by geological survey results (Manatschal, 1999; Oohashi et al., 2012).

485 Therefore, we subjected both the initial  $\sigma_0$  values and the fitted  $\sigma_{ss}$ - $X_{Gr}$  curve of our study 486 and those of previous experiments under higher static pressure conditions (Chen et al., 2017) for 487 further discussion. The  $\sigma_0$  values showed similar enhancement with increasing  $X_{\rm Gr}$  to  $\sigma_{\rm ss}$  values (from  $\sim 10^{-10}$  S/m to  $\sim 10^{3}$  S/m). Under static compaction, the higher the normal stress, the lower 488 489 the threshold value (X<sub>c</sub>, 6.0% under up to 300 MPa vs. 11.3% under 2 MPa), while the sheared 490 specimen has the lowest  $X_c$  value (3.4%). Therefore, we posit two enhancement factors of  $X_c$ 491 (threshold value) and electrical conduction, i.e., shear deformation (a red shadow in Figure 8) 492 and static compaction (a blue shadow in Figure 8). Meanwhile, deep Gr flakes can remain stable 493 up to crustal depth (Selway, 2013). Therefore, assuming high pressure and temperature 494 conditions, which are common in fault conditions, high  $\sigma$  values can be readily achieved in 495 natural faults when  $X_{Gr}$  reaches 3.4%, given an extremely high growth rate across the critical 496 value (upward pathway labeled by red dashed arrow in Figure 8).

497 The conductive properties of the upper crust can be affected by many electrical conduction 498 factors, such as pressure, temperature, graphite (Frost et al., 1989; Nover et al., 1998), saline 499 fluid (Guo & Keppler, 2019; Sinmyo & Keppler, 2017), sulfide (Watson et al., 2010), or partial 500 melting (Chen et al., 2018). Furthermore, the coexistence of interconnected melt/fluids and Gr 501 veins also seem to provide the best explanation in several low resistive fault zone derived from 502 MT profiles (Wannamaker et al., 2002; Yu et al., 2020; Zhao et al., 2012) because the 503 interconnected saline fluid improves the Gr-vein conduction (depositing hydrothermal Gr or 504 promoting conductivity) (Kirilova et al., 2018b; Oohashi et al., 2012). To disentangle the effect 505 of these mechanisms, we need to apply the control variable method, i.e., separately study these 506 factors one by one to clarify their effects and finally summarize them and bring them together for 507 comparison. Our work currently focuses only on the effect of Gr-bearing conductive networks on 508 high conductivity anomalies at fault zones. Further experimental investigations at high 509 temperatures and high pressure are necessary to constrain the mechanical and electrical 510 conduction mechanisms of high conductivity brittle-to-ductile shear zones.

511

#### 512 4.2 Electrical conductivity variations at initiation of frictional slip

513 4.2.1 Electrical conductivity jump as a potential indicator for fault slip

514 The results of our mechanical–electrical experiments at the millimeter scale show that the  $\sigma$ -

515 jump phenomenon of Qz–Gr mixtures with > 4.6 vol.% Gr (> 4 wt.% Gr) occurred before

516 steady-state frictional slip, and even before the peak friction (see Figures 4a–4c vs. Figures 4d–4f, 517 and the inset of Figure 9). This suggests that this jump can appear before steady-state fault slip, 518 i.e., initiated frictional slip that may generate potential electromagnetic anomaly signals (the red 519 area in Figure 9). In the following, we apply this phenomenon to natural Gr-enriched fault zones. 520 According to the classic earthquake nucleation theory, quasi-static or pre-slip occurs in the 521 local area of a fault (i.e., the nucleation zone) before an earthquake (e.g., Dieterich, 1992; Rubin 522 & Ampuero, 2005). With the expansion of the slip region, the fault appears to have irreversible 523 dynamic expansion as it reaches the critical nucleation scale. Therefore, based on the above 524 experimental observations, it is possible to detect an initiated slip of the fault by monitoring the 525 change in the  $\sigma$  at the nucleation zone of the fault (see the abnormal electrical signals detected by 526 the electrical resistivity stations in Figure 9). Besides the pre-slip, continuous or periodic creep 527 (slow slip) of faults can also cause variations in electrical conductivity values. However, we 528 cannot determine the cumulative slip displacement of the fault zone by electromagnetic data 529 monitoring. If the slip has accumulated too large, that will lead to the development of a more 530 mature shear zone structure (similar to the late stage in our experiment). The abrupt change in the 531 electrical conductivity will hardly be observed. Therefore, thus far, for actual applications, our 532 experimental results may not provide evidence for distinguishing fault creeps.

533

#### 534 4.2.2 Limitations and future development

Although this study is the first attempt to constrain the electrical response of carbon-bearing
fault gouges under dynamic friction conditions, our conclusions have some limitations.

537 Firstly, the  $\sigma$  measurements used a single-frequency direct current method. However, the 538 observed data of seismic georesistivity stations in the field cover a wide frequency range, which 539 can reflect the electrical structure over various depth ranges (see the electrical current pathways 540 at different depths in Figure 9). Although we sacrificed the accuracy of the electrical 541 measurements, the transient electrical conductivity and the corresponding characteristic 542 displacements of sheared fault gouge can be obtained in this study. In fact, our experimental data 543 obtained based on the DC single-frequency method are close to those measured by the AC 544 impedance spectroscopy on the same sheared Gr-Qz mixtures at the same conditions (Han et al., 545 2019). We believe that the precision of the experimental data meets the requirements of this study. 546 Secondly, only the specimens with > 17 vol.% Gr (or > 15 wt.% Gr) under our experimental 547 conditions (i.e., low normal stress, room temperature, and dry conditions) exhibited the  $\sigma$ -jump 548 phenomenon prior to the peak friction (Figure 4f). Fluid is widely present in nature fault zones 549 and plays important roles in various aspects (Hickman et al., 1995). Previous studies also 550 proposed that electrical anomalies in the brittle shallow fault zone are caused by pore fluid 551 within rock fractures (Du, 2011; Park et al., 1993; Zhao & Qian, 1994). Against this background, 552 the electromagnetic anomaly signals due to Gr interconnection revealed in this study may be 553 limited to anhydrous fault environments, such as deep cataclasites with low porosities (bold blue 554 dashed line across the preslip zone in Figure 9). Temperature and pressure conditions may also 555 affect the results. Our previous experiments under static conditions revealed that elevated 556 pressure can facilitate the grain-boundary Gr conduction and significantly reduce the threshold 557 value (i.e., from 11.3 to 6.0 vol.% as pressure increases to 300 MPa, Figure 8). Taking this effect 558 into account, the electromagnetic anomaly is expected to be generated under short shear 559 displacement and/or lower threshold value. As indicated earlier, a natural Gr-bearing fault may 560 have Gr content up to 12 wt.% (Manatschal, 1999; Oohashi et al., 2012). At elevated 561 temperatures, plastic deformation mechanisms come to play an increasing role, and at some point, the silicate minerals begin to exhibit semiconductor behavior (e.g., >200-300 °C, Selway, 2013). 562 563 All these processes can enhance the conductivity and affect the threshold values (Wang et al., 564 2013). To investigate these effects, systematical experiments using high-temperature and high-565 pressure deformation apparatuses such as the Paterson rig are planned in the future.

566

#### 567 **5 Conclusions**

We designed a rotary-shear setup to monitor the transient electrical response of synthetic dry quartz (Qz)–graphite (Gr) mixtures in the shear-parallel direction during progressive slip. Long-displacement friction experiments (0.9–4.2 m) were performed at fixed normal stresses, slip rate, and N<sub>2</sub>-flushing atmosphere.

572 (1) Graphite volume fraction  $(X_{Gr})$  and slip displacement had important effects on the 573 frictional coefficient ( $\mu$ ) and electrical conductivity ( $\sigma$ ) of the mixture. The steady-state frictional 574 coefficient ( $\mu_{ss}$ ) of the mixtures with low  $X_G$  (< 13.6 vol.%) maintained high levels of frictional 575 strength ( $\mu = 0.54$ –0.80), while the mixtures with high  $X_G$  (> 13.6 vol.%) showed remarkable slip 576 weakening behavior where the  $\mu_{ss}$  decreased with the increase of  $X_{Gr}$ . The  $\sigma$  of the mixtures with 577  $\geq$  4.6 vol.% X<sub>Gr</sub> abruptly increased ( $\sigma$  jump) with limited shear displacement; some  $\sigma$  jumps 578 occurred even before the peak friction (as low as 0.2 mm). With continued shear, the steady-state 579 electrical conductivity ( $\sigma_{ss}$ ) increased by more than seven orders of magnitude when  $X_{Gr} > 3.4$ 580 vol.%. The post-mortem microstructures revealed that the high  $\sigma_{ss}$  observed in the intermediate 581 Gr content specimens (3.4-13.6 vol.%) was associated with an *ad-hoc* fabric (graphite-cortex 582 clasts, GCCs) present in the principal slip zone. Excess Gr flakes can fill the pores and help 583 develop Gr-coated mechanically lubricated surfaces.

584 (2) The percolation model can capture the relationship between the  $\sigma_{ss}$  and  $X_{Gr}$  of the Qz–Gr mixture. However, the percolation theory required adaption in the future to incorporate the 585 586 anisotropic transport structure more accurately. Compared with the observations of 587 magnetotelluric (MT) surveys, our experimental results revealed that dry sheared fault rocks 588 containing 5.4–8.1 vol.% Gr may be responsible for the highly conductive anomalies at shear 589 zones. Furthermore, considering the effect of high normal stress from our previous study (Chen 590 et al., 2017), the high electrical conductivity ( $\sigma$ ) in the natural fault may also be achieved when 591 the  $X_{Gr}$  is as low as 3.4 vol.%.

(3) The observed  $\sigma$ -jump phenomenon suggests that an initiated slip in the carbonaceous shear zone may generate potential electrical anomaly signals that can be detected by electromagnetic stations. The electrical anomaly due to Gr interconnection may be limited to anhydrous fault environments. Further experimental investigations for fluid-bearing specimens at high temperature and high pressure, applying the frequency sweep method, are required to constrain mechanical and electrical conduction mechanisms of shear zones.

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605

# 606 Open Research

- 607 All the data used for this study have been made available through Mendeley Data
- 608 (<u>https://doi.org/10.17632/p4bs2tb58h.1</u>) (Chen et al., 2023).

609

Figure 1. Illustration of the friction–conductivity testing system at the Institute of 610 Geology, China Earthquake Administration (IGCEA). (a) A low- to high-velocity 611 rotary shear apparatus, which provides a frictional environment. (b) Appearance of 612 the 4-wire (i.e., using wires 1–4) experimental assembly amplified from (a). The two 613 614 small figures in the upper right corner show a non-specimen assembly and a specimen-bearing assembly before prepressing. (c) The electrical conductivity 615 measurement apparatus, i.e., Keithley low-level sensitive and specialty instruments. 616 They contain three models adapted to different electrical resistance ranges of Gr-617 bearing specimens (the detailed settings are presented in Table 1). (d) Diagram of a 618 Gr-bearing specimen assembly adapted to transient electrical conductivity 619 measurement by the 2- or 4-wire setups. An arced arrow indicates the shear direction. 620  $P_{\rm n}$  indicates constant normal stress. (e) A schematic representation of the specimen 621 622 and sections chosen for microstructural analysis (see Figure 5).

623

Figure 2. Frictional coefficient and electrical conductivity for the Gr–Qz mixtures 624 sheared at a normal stress of 2 MPa. (a-b) Frictional coefficient vs slip displacement. 625 626 (c-d) Electrical conductivity vs slip displacement. The slip displacement is plotted 627 on (panels a and c) logarithmic and (panels b and d) linear scales. The percentages in the legend indicate the Gr contents of the specimens in weight ratio.  $D_{\sigma ch}$ ,  $D_{\mu ss}$ , and 628 629  $D_{\sigma ss}$  denote the characteristic displacements at which the electrical conductivity initiates significant changes and at which the evolution of friction coefficient and 630 electrical conductivity reach steady states, respectively (marked as yellow diamond, 631 pentagram, and square symbols on each curve, respectively). We note that the final 632 jump and hold-time data ( $v_e < 0.6 \text{ mm/s}$ ) of all experimental values were removed. 633

634

Figure 3. Frictional coefficient and electrical conductivity for the Gr–Qz mixtures
sheared at a normal stress of 5 MPa. Details are the same as those in Figure 2.

637

638 Figure 4. Comparison of experimental data related to characteristic displacements. 639 (a-f) Electrical conductivity and friction coefficient plotted against displacement data at displacements between  $D_{\sigma ch}$  (diamond labels) and  $D_{\sigma ss}$  (square labels) are 640 highlighted by bold solid lines. Gr fractions correspond to (panels a and d) 4-10 641 wt.%, (panels b and e) 9-12 wt.%, and (panels c and f) 15-50 wt.%. Further details 642 643 are the same as those in Figure 2.  $D_0$  denotes the characteristic displacements at which the specimen initiates shear deformation (marked as a circle of the same color 644 645 on each curve). (g-h) Variations of  $D_{\sigma ch}$ ,  $D_{\sigma ss}$ , and  $D_{\mu ss}$  vs Gr fraction in the Gr-Qz mixtures sheared under normal stresses of 2 and 5 MPa. Definitions of the symbols 646 are the same as those in Figure 2. 647

648

**Figure 5**. BSE images of the Gr-bearing specimens sectioned parallel to the axis of cylindrical host blocks. (a) 0 wt.% Gr, (b) and (e) 3 wt.% Gr, (c) and (f) 9 wt.% Gr, and (d) 25 wt.% Gr, respectively. The specimen number, final slip displacement, Gr content, and normal stress of each experiment are shown above the images. Yellow dotted lines in panels b and c show boundaries of the three layers with different degrees of shear deformation (Layers I to III with ascending shear deformation). Gr:graphite; Qz: Quartz; ER: Epoxy Resin.

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**Figure 6.** Microphotographs of deformed and undeformed layers for specimen. It is an unepoxied piece of the gouge layer recovered after the run LHV2429 (12 wt.% Gr at 2 MPa). (a) Geometry of the specimen and location of the spot for SEM. (b–c) SE images of the upper surface of the slip-localized zone (akin to the top surface of Layer I in Figures 5b–5c). (d–f) BSE image of the weakly or undeformed zone (akin to Layer III in Figures 5b–5c).

663

**Figure 7**. Schematic diagram of the fitting  $\mu_{ss}$ - $X_{Gr}$  relationship (solid blue line), the fitted  $\sigma_{ss}$ - $X_{Gr}$  relationship (solid red line), and four classes of frictional textures for Gr–Qz mixtures. The cartoon shows the proposed graphite–cortex clast (GCC) fabric.

667

Figure 8. Electrical conductivity vs.  $X_{Gr}$ . Experimental values of the initial electrical 668 conductivity ( $\sigma_0$ ) at 2 MPa (solid red line) and 5 MPa (solid blue line) and a fitting 669 curve (solid black line) of the steady-state electrical conductivity ( $\sigma_{ss}$ ) plotted against 670 671 the Gr volume percentage  $(X_{Gr})$  for the Qz–Gr mixtures. The grey dashed line shows the conductive trend of identical mixtures in this study under uniaxial compaction at 672 0.1-300.0 MPa (Chen et al., 2017). The orange regime (0.100-0.005 S/m) indicates 673 the general range of highly conductive field values in shear zones derived from 674 magnetotelluric surveys (e.g., Pous et al., 2004; Ritter et al., 2005; Wannamaker et 675 al., 2002; Zhao et al., 2012). 676

677

**Figure 9.** Schematic diagram of an electromagnetic station layout across a carbonaceous fault zone. The inset indicates the variations of shear stress ( $\tau$ ) and electrical conductivity ( $\sigma$ ) of the initiated slip zone.

Gr wt.%	$R_{\rm E}$ range	Measurement model	Accuracy
0–3%	> 200 GΩ	4-wire setup (using wires 1–4), $R_E = E/I$ , where constant DC current ( <i>I</i> ) is supplied by the 6221 AC and DC Current Source and electrical potential ( <i>E</i> ) is measured by the 6514 System Electrometer.	±0.46%
4–12%	200 Ω–200 GΩ	2-wire setup (using wires 1–2), where $R_{\rm E}$ is directly measured by the 6514 System Electrometer.	±1.50%
15-100%	< 200 Ω	4-wire (using wires 1–4), $R_{\rm E}=E/I$ , where I is supplied by the 6221 AC and DC Current Source and <i>E</i> is measured by the 2182A Nanovoltmeter.	±0.42%

**Table 1**. Summary of electrical conductivity measurement models.

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No	Spacimon	Gr%	error	$D_{\mu m ss}$	$\mu_{ m ss}$	$\mu_{ m ss}^{\scriptscriptstyle (+)/(-)}$	$D_{\sigma { m ch}}$	$D_{\sigma  m ss}$	$\sigma_0$	$\sigma_{ m ss}$	$\sigma_{ m ss}^{\scriptscriptstyle (+)}$	$\sigma_{ m ss}^{\scriptscriptstyle (-)}$
INO.	Specifien	vol.%	±vol.%	m			m	m	S/m	S/m	S/m	S/m
					Series	I (2 MPa)						
LHV2051	100 wt.% Qz	0.00	0.00	nd	nd	nd	nd	0.894	1.54×10 <sup>-7</sup>	4.38×10 <sup>-11</sup>	3.56×10 <sup>-11</sup>	1.96×10 <sup>-11</sup>
LHV2416	3 wt.% Gr+97 wt.% Qz	3.44	0.08	2.229	0.79	0.01	nd	2.89×10 <sup>-4</sup>	1.10×10 <sup>-9</sup>	8.68×10 <sup>-11</sup>	9.39×10 <sup>-11</sup>	4.51×10 <sup>-11</sup>
LHV1374	4 wt.% Gr+96 wt.% Qz	4.58	0.09	0.397	0.80	0.03	0.500	1.161	1.80×10 <sup>-10</sup>	1.40×10 <sup>-3</sup>	1.16×10 <sup>-3</sup>	6.34×10 <sup>-4</sup>
LHV1372	5 wt.% Gr+95 wt.% Qz	5.72	0.11	0.566	0.54	0.01	0.233	0.788	5.53×10 <sup>-10</sup>	5.90×10 <sup>-3</sup>	5.05×10 <sup>-3</sup>	2.72×10 <sup>-3</sup>
LHV1373	6 wt.% Gr+94 wt.% Qz	6.85	0.12	0.525	0.62	0.04	0.158	1.048	3.59×10 <sup>-10</sup>	0.089	0.087	0.044
LHV1371	9 wt.% Gr+91 wt.% Qz	10.23	0.16	0.508	0.78	0.00	7.01×10 <sup>-4</sup>	0.206	6.93×10 <sup>-10</sup>	0.345	0.256	0.147
LHV2052	10 wt.% Gr+90 wt.% Qz	11.35	0.17	nd	nd	nd	0.004	0.113	2.28×10 <sup>-10</sup>	0.018	4.36×10 <sup>-3</sup>	3.50×10 <sup>-3</sup>
LHV2429	12 wt.% Gr+88 wt.% Qz	13.57	0.20	nd	nd	nd	1.94×10 <sup>-4</sup>	0.004	0.268	4.367	2.267	1.492
LHV1380/2050	15 wt.% Gr+85 wt.% Qz	16.89	0.23	0.321	0.62	0.03	1.79×10 <sup>-4</sup>	0.009	1.679	1.473	0.060	0.058
LHV2418	25 wt.% Gr+75 wt.% Qz	27.74	0.31	0.150	0.19	0.01	3.89×10 <sup>-4</sup>	0.003	32.59	54.61	9.211	7.882
LHV2419	50 wt.% Gr+50 wt.% Qz	53.53	0.37	0.339	0.11	0.00	4.39×10 <sup>-4</sup>	0.003	171.0	421.5	31.68	29.47
					Serie	es II (5 MPa	ı)					
LHV2057	100 wt.% Qz	0.00	0.00	nd	nd	nd	nd	0.170	8.15×10 <sup>-8</sup>	1.78×10 <sup>-11</sup>	1.13×10 <sup>-12</sup>	1.06×10 <sup>-12</sup>
LHV1823/2054	3 wt.% Gr+97 wt.% Qz	3.44	0.08	0.361	0.85	0.03	nd	0.594	2.70×10 <sup>-10</sup>	1.57×10 <sup>-10</sup>	2.00×10 <sup>-11</sup>	1.77×10 <sup>-11</sup>
LHV1375 <sup>*</sup>	6 wt.% Gr+94 wt.% Qz	6.85	0.12	0.413	0.80	0.06	0.175	0.743	8.65×10 <sup>-9</sup>	0.011	0.023	7.29×10 <sup>-3</sup>
LHV2055	9 wt.% Gr+91 wt.% Qz	10.23	0.16	0.407	0.84	0.12	7.94×10 <sup>-4</sup>	0.057	5.64×10 <sup>-4</sup>	0.359	0.085	0.069
LHV2056	15 wt.% Gr+85 wt.% Qz	16.89	0.23	0.337	0.74	0.03	5.98×10 <sup>-4</sup>	0.010	13.57	54.37	12.00	9.828
LHV2053	100 wt.% Gr	100	0.00	0.283	0.10	0.00	0.000	0.000	1095	1077	22.19	21.74

**Table 2**. Summary of the obtained experimental data.

684 Gr: graphite; Qz: quartz;  $\mu_{ss}$ : steady-state frictional coefficient;  $\mu_{ss}^{(+)(\cdot)}$ : standard deviation of  $\mu_{ss}$ ;  $D_{\mu ss}$ : slip displacement as  $\mu$  achieved  $\mu_{ss}$ ;  $\sigma_0$ : initial electrical conductivity;  $\sigma_{ss}$ : steady-state

685 electrical conductivity;  $\sigma_{ss}^{(+)}/\sigma_{ss}^{(-)}$ : standard deviation of  $\sigma_{ss}$ ;  $D_{\sigma ch}$ : slip displacement as conductivity initiated logarithmic change;  $D_{\sigma ss}$ : slip displacement as  $\sigma$  achieved  $\sigma_{ss}$ , and nd indicates that the

686 parameter could not be determined due to erratic frictional behavior.

**Table 3.** Fitted parameters of Equation 2 for the relationships between  $\mu_{ss}$  and  $X_{Gr}$ . Fitted parameters proposed by

688 Oohashi et al. (201	<ol><li>13) are also listed</li></ol>
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$\mu_{ m Qz}$	$\mu_{ m Gr}$	$X_{ m cw}$	S	Data source and slip rate
0.73	0.10	0.223	8.97	This study; 1 mm/s
0.65	0.09	0.128	1.82	Oohashi et al., 2013; 0.2 mm/s
0.57	0.09	0.118	1.56	Oohashi et al., 2013; 21-56 mm/s

<sup>689</sup>  $\mu_{Qz}$  and  $\mu_{Gr}$ :  $\mu$  of pure Qz and Gr, respectively;  $X_{cw}$ : critical slip-weakening fraction for  $X_{Gr}$ ; S: slope parameter.

690

691

**Table 4.** Fitted parameters of Equation 3 for the relationships between  $\sigma_{ss}$  and  $X_{Gr}$ . Fitted parameters proposed by

693 Chen et al. (2017) are also listed.

X <sub>c</sub>	$\sigma_{ m Oz}$	$\sigma_{ m Gr}$	- 0	r Data source and experiment condition	
vol.%	S/m	S/m	u	7	Data source and experiment conditions
11.3	$2.28 \times 10^{-10}$	>171.0			Initial compaction at 2 MPa
6.8	$2.70 \times 10^{-10}$	1095.0			Initial compaction at 5 MPa
3.4	$1.11 \times 10^{-10}$	1077.0	1.29	3.36	Post-shear at 2–5 MPa
6.0	$1.32 \times 10^{-10}$	889.6	3.37	2.53	Chen et al., 2017; uniaxial compression

694  $\alpha$ : geometric factor of the conductor (Gr); r: nondimensional parameter;  $X_c$ : threshold value of  $X_{Gr}$ ;  $\sigma_{Qz}$ : electrical

695 conductivity of the insulating matrix (Qz);  $\sigma_{Gr}$ : electrical conductivity of the conductor (Gr).

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945

Figure 1.



Figure 2.



Figure 3.



Figure 4.



Figure 5.



Figure 6.

# LHV2429 (1.84 m) 12 wt.% Gr-2 MPa

Layer I Intensive Foliation



Layer III Non-Foliation





Figure 7.





Figure 8.



Figure 9.

# Surface fault trace

