Linking thermal and rate behavior with fabric and mineralogy of clayey shear zones: Experimental investigation on the El Forn landslide (Andorra)

Carolina Seguí^{a,*}, Esperança Tauler^b, Tomasz Hueckel^c, Manolis Veveakis^c

^aRWTH Aachen University, Aachen, Germany ^bUniversity of Barcelona, Barcelona, Spain ^cDuke University, Durham, NC, USA

Abstract

Thermal triaxial tests were carried out on remolded material of core samples of the shear zone of a deep-seated landslide, the El Forn landslide (Andorra). With the thermal triaxial tests, we have obtained thermal and rate sensitivities of the material outside and inside the shear band. Moreover, micro-scale tests were carried out, such as XRPD, SEM-EDS, MicroCT, and plasticity index. The results show that the material inside the shear band has higher thermal sensitivity than outside the shear band. These results are related to the material's fabric, porosity, and plasticity index. The rate sensitivity value is the same for the samples outside and inside the shear band, being able to correlate the rate sensitivity with the mineralogy content of the material. This study, thus, aims to understand the mechanical behavior of the material within a shear zone by correlating the thermal and rate sensitivity with the microstructure and mineral phases present in the material.

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^{*}Corresponding author: C. Seguí

Email address: segui@gut.rwth-aachen.de (Carolina Seguí)

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1 1. Introduction

This paper presents the behavior of clayey materials, outside and inside 2 the shear zone, of a deep-seated landslide by linking the thermal and rate 3 sensitivities with the fabric and mineralogy of the material. The effect of friction/shearing in a clayey layer with continuous creeping changes the orientation of the mineral particles, thus affecting the material's mechanical behavior by decreasing its friction coefficient and increasing the weakness of the material (Anderson, 1980; Lachenbruch, 1980; Mase & Smith, 1985; 8 Rice, 2006; Vardoulakis, 2002b; Voight & Faust, 1982). Moreover, changes 9 in the loading rate on the material also affect the mechanical behavior of 10 the shear layer (Song et al., 2014; Wang et al., 2017; Rattez & Veveakis, 11 2020; Rattez et al., 2018), depending on its mineralogy content. The effect 12 of thermal and loading rate behavior of geomaterials, especially in clays, has 13 been explored in the fields of fault mechanics (i.e., earthquakes) (Rice, 2006; 14 Chambon et al., 2002; Tesei et al., 2012; Kohli & Zoback, 2013; Veveakis 15 et al., 2010; Alevizos et al., 2014; Veveakis et al., 2014; Poulet et al., 2014), 16 nuclear waste disposal (Hueckel & Baldi, 1990; Hueckel & Borsetto, 1990; 17 Hueckel & Pellegrini, 1991, 1992; Del Olmo *et al.*, 1996; Hueckel *et al.*, 2009; 18 Monfared et al., 2014; Gens et al., 2018; Laloui, 2001), and landslides (Var-19 doulakis, 2002a,b; Veveakis et al., 2007; Ciantia & Hueckel, 2013; Seguí et al., 20 2020; Seguí & Veveakis, 2021; Goren & Aharonov, 2007), as well as energy 21 applications (Veveakis & Regenauer-Lieb, 2015; Laloui & Cekerevac, 2003; 22

²³ Sari *et al.*, 2020).

In fault mechanics and earthquake engineering, the effect of rate sensi-24 tivity of the material has been tested to understand the mechanics of the 25 gauge material (Chambon et al., 2002; Tesei et al., 2012; Kohli & Zoback, 26 2013; Sulem et al., 2011). In nuclear waste disposal, the effect of thermal 27 loading has been an important parameter in studying the material's behav-28 ior due to the low permeability of some clays and to understanding how the 29 material responds when is under very high temperatures (Hueckel & Baldi, 30 1990; Hueckel & Borsetto, 1990; Hueckel & Pellegrini, 1991, 1992; Del Olmo 31 et al., 1996; Hueckel et al., 2009; Monfared et al., 2014; Gens et al., 2018). 32 In landslides, the effect of rate sensitivity has been considered for the shear 33 band material due to the changes in groundwater levels (seasonal variations, 34 reservoir levels, etc.). Moreover, several authors consider the effect of tem-35 perature on landslides due to shearing movement increases the temperature 36 in the material due to friction. The increase of temperature in the material 37 modifies the friction coefficient of the shear band's material (Vardoulakis, 38 2002b; Veveakis et al., 2007; Pinyol & Alonso, 2010; Alonso et al., 2016; 39 Seguí et al., 2020; Seguí & Veveakis, 2021, 2022), as well as the effects of 40 degradation of the material due to weathering, which leads to slope insta-41 bilities (Ciantia & Hueckel, 2013; Cecinato et al., 2010; Cecinato & Zervos, 42 2012). 43

This study focuses on the role of thermal sensitivity due to the fabric of the material, especially in clays or clayey materials. Previous studies have been performed, to have a better understanding of the behavior of clayey materials. In particular, Seguí & Veveakis (2021) presented the results of

thermal sensitivity in black shales of the Silurian, outside and inside the 48 shear band, of a deep-seated landslide. The results showed different values 40 of thermal sensitivity, being higher inside the shear band where the phyl-50 losilicates are completely aligned face-face (Seguí et al., 2021). In addition, 51 Del Olmo et al. (1996) analyzed the thermomechanical properties of Span-52 ish and Boom clays at temperatures close to nuclear waste disposal, finding 53 that the material is thermal softening and strain hardening. In that study, 54 Del Olmo et al. (1996) already discussed that the effect of the microstruc-55 ture could affect the microfractures and dehydration of the material, leading 56 to those thermomechanical results. Later, Hueckel et al. (2009) performed 57 thermal tests on Spanish clay obtaining thermal softening and plastic strain 58 hardening of the material (i.e., reduction of strength with increasing tem-59 perature). In their study Hueckel et al. (2009) discuss that the decrease of 60 internal friction while the increasing temperature is related to the contacts 61 of the particles, as it affects the water absorbed by the particles depending 62 on how they are connected (face-face, face-edge, edge-edge). 63

In addition, the present work also analyzes the interplay between the load-64 ing rate sensitivity of a clayey material related to the mineralogy content. 65 Previous studies have presented results of the behavior of clavey materials 66 under different loading rates. Seguí & Veveakis (2021) presented the results 67 of rate sensitivity for black shales of the Silurian outside and inside the shear 68 band of a deep-seated landslide. They obtained the same values of rate 69 sensitivity for all the samples tested, thus suggesting that the rate sensitiv-70 ity depends on the sample's mineralogy rather than the orientation of the 71 mineral particles. In the mid-1970s, Leinenkugel (1976) performed velocity

stepping tests on remolded kaolin clay, obtaining the rate hardening of the 73 material and its law. Later, Ikari et al. (2009) performed laboratory tests 74 on synthetic montmorillonite gauge through velocity stepping in a biaxial 75 apparatus, obtaining as well rate hardening of the material. Finally, Samuel-76 son & Spiers (2012) performed laboratory tests in illite simulated fault gouge 77 through velocity stepping in a direct shear machine, obtaining rate hardening 78 of the material (i.e., strength increase of the material, when the loading rate 79 increases). 80

Several studies have presented constitutive models combining the effects 81 of rate hardening and thermal softening. Firstly, Hueckel & Borsetto (1990) 82 postulated the constitutive equations of the plastic behavior of clay and shales 83 from the experiments performed previously by Hueckel & Baldi (1990). These 84 constitutive equations express the counterbalancing effects of thermal soften-85 ing and plastic strain hardening. Afterward, Vardoulakis (2002b) presented 86 a constitutive model of the thermo-viscoplastic coupling in clavey gouges. 87 Vardoulakis (2002b) showed the results of thermal softening from Hicher 88 (1974), rate hardening of Leinenkugel (1976), and explained the importance 80 that both effects must counterbalance. Later, Veveakis et al. (2007), Seguí 90 et al. (2020), and Seguí & Veveakis (2021) continued this line of Vardoulakis 91 (2002b) in deep-seated landslides strengthening the thermal sensitivity and 92 rate hardening coupling and proving the assumption by performing labora-93 tory experiments on the gauge of a deep-seated landslide. Moreover, Hueckel 94 et al. (2009) presented an in-depth study of the thermal failure in satu-95 rated clays combining mathematical modeling with experimental data. The 96 authors, again, showed the importance of counterbalancing the effects of 97

⁹⁸ thermal softening and rate hardening of the clayey material.

The validation of the aforementioned counterbalanced constitutive laws 99 has been presented and discussed previously by the authors (Seguí & Ve-100 veakis, 2021). They presented experimental results of thermal and rate sen-101 sitivities of the Silurian black shales of the El Forn landslide (Andorra). 102 Moreover, the authors have previously performed on the same Silurian shales 103 mineralogical tests by X-Ray Powder Diffraction (XRPD), textural analysis 104 by Scanning electron microscope with energy-dispersive X-ray spectroscopy 105 (SEM-EDS), porosity by Micro X-ray computed tomography scanner (Mi-106 croCT), and plasticity index (Seguí et al., 2021). However, the link between 107 the thermal and rate sensitivity behavior with the material's mineral con-108 tent and the internal structure has not been examined and discussed yet. 109 Therefore, this paper presents an in-depth study of experimental tests in a 110 thermal-triaxial machine on the same black shales of the Silurian period from 111 core samples inside and outside the shear band of the El Forn landslide (An-112 dorra), and the micro-scale tests performed as well (Seguí *et al.*, 2021). We 113 have merged all the results and presented them in this paper to understand 114 and link the differences obtained from the thermal and rate sensitivity tests 115 with the fabric and mineralogy of the samples. This paper aims to shed light 116 on the mechanisms present in shear bands containing clavey materials and 117 understand why the material behaves as softening or hardening when the 118 temperature and the loading rate change. 119

120 2. Materials

In this work, we study the core samples of the inside and outside the shear 121 band of the lobe Cal Ponet-Cal Borronet inside the large El Forn landslide 122 (Andorra) (Fig. 1 an S10 borehole). This lobe is approximately $1Mm^3$ 123 of rock mass sliding as a rigid block (i.e., translational/rotational) with an 124 average velocity of 2cm/year (Corominas *et al.*, 2014) (Fig. 1b). The samples 125 we test in this study were preserved from the S10 borehole performed at the 126 Cal Ponet-Cal Borronet lobe in 2007 (Fig. 1c). In Fig. 1c can be seen that the 127 samples we study are fully saturated in water, indicated by the information 128 from piezometers installed in the borehole (EuroconsultSA, 2017). The shear 129 band of the lobe and its surroundings are formed by black shales from the 130 Silurian (Clariana, 2004). The core samples studied are located between 27 131 and 30m depth (Figure 1b,c), being its shear band located at 29-29.5m depth 132 (Fig. 1b,c) (Seguí et al., 2021). In this study, we will divide each sample 133 every half a meter of the vertical depth, thus at every 0.5m, characterizing six 134 samples to see the evolution in-depth of the mineralogy, orientation, porosity, 135 and plasticity index. 136

The core samples have been studied at the micro-scale to determine their mineralogy composition, the fabric (i.e., the orientation of the minerals), the porosity, and its plastic and liquid limits. At the meso-scale, we determine the critical-state line of the material (black shales of the Silurian), the friction coefficient at the critical state, and the thermal and rate sensitivities.



Figure 1: The deep-seated landslide of El Forn in Andorra. (a) Google Earth image highlighted the large El Forn landslide (red) and the Cal Ponet-Cal Borronet lobe (purple), with the S9 and S10 boreholes. (b) Extensometer displacement data profile (in-depth) of the S10 borehole. The data is from April to June 2017 (EuroconsultSA, 2017). (c) Core samples preserved of the S10 borehole performed in 2007, including the shear band of the Cal Ponet-Cal Borronet lobe and its center highlighted in a red rectangle. The samples tested in this study are highlighted by a purple rectangle.

¹⁴² 3. Micro-scale characterization of the shearing zone material

In this section, we explain the technical features of the micro-scale tests performed on the black shales of the Silurian from the Cal Ponet - Cal Borronet lobe. The micro-scale tests performed on the samples are: XRPD to obtain the mineralogy, SEM-EDS to see the fabric and orientation of the mineralogy, MicroCT for the porosity, and Plasticity Index as the mechanical property of the samples.

149 3.1. X-Ray Powder Diffraction

Samples studied in XRPD needed to be crushed in an agate mortar until 150 the rock reduces its particle size below $40\mu m$, ending as fine powder that does 151 not scrape between the fingers. XRPD data were collected with Panalyti-152 cal X'Pert PRO MPD X-ray diffractometer, with monochromatized incident 153 $CuK\alpha 1$ radiation at 45kV and 40mA, equipped with a PS detector with an 154 amplitude of 2.113° . Patterns were obtained by scanning randomly-oriented 155 powder particles from 4° to 80° (2 θ). Datasets were obtained using a scan 156 time of 50 seconds at a step size of 0.017° (2 θ) and a variable automatic 157 divergence slit. The identification of minerals was achieved by comparing 158 XRPD with the ICDD database (2007 release) using Diffrac plus Evaluation 159 software (Bruker, 2007). Quantitative mineral phase analyses were obtained 160 by full refinement profile using XRPD and the software TOPAS V4.2 (2009) 161 (Coelho, 2000). 162

3.2. Scanning Electron Microscope with Energy-Dispersive X-Ray Spectroscopy
 To study samples in SEM-EDS, we have created thin polished thin sec tions from the core samples. The sample preparation procedure of a thin

section includes cutting a $1000mm^3$ cubical or prismatic sample from a cylin-166 drical core of undisturbed material retrieved at a depth between 27m to 30m167 (shear band). A cubical sample is first included in epoxy resin and then at-168 tached to a glass sample holder -on one side-, to cut a slice of $50\mu m$ thick on 169 which SEM observations will be performed. Finally, a metallographic polish-170 ing -under dry conditions-, is carried out to obtain the flat surface needed for 171 EDS analyses. Morphology and microtextural features of the studied sam-172 ples were examined with Nikon Eclipse LV100 POL microscope and ESEM 173 Quanta 200 FEI, XTE 325/D8395 scanning electron microscope with energy-174 dispersive X-ray spectroscopy (SEM-EDS). 175

176 3.3. Micro X-Ray Computed Tomography Scanner

MicroCT tests were performed on the studied core samples. Multiple 177 tests have been carried out to ensure the representativeness of the samples 178 and repeatability of the results. The porosity of the studied samples was 179 examined on chips of original core rock (order of cm) with a Nikon XTH 180 225 ST, which is a high-resolution X-Ray computed tomography scanner. 181 With this scanner, 2-D and 3-D images of inside and outside the sample 182 are obtained by projection of an X-Ray beam through the sample and the 183 sample's interaction with the beam via radiography/X-Ray image. The X-ray 184 source used in the tests performed is the 225kV UltraFocus Reflection Based 185 Signal and Tungsten Target (Spot Size $3\mu m$) with a Max Power of 200W. 186 The detector used is the Perkin Elmer 1620 AN3 CS CT. The X-ray filter 187 used is a 0.5mm thick copper filter. We have used the Avizo Software (TJP, 188 2015) to post-process the 2-D Micro-CT images. The 3-D reconstruction 189 and the quantification of the matrix and the porosity, separately, have been 190

performed by segmenting the 2-D images (i.e., separating the pores from the
matrix using a gradient threshold method).

Once the segmentation is successful (i.e., the contacts between the matrix 193 and the pores are well defined as well as the contour of the sample from the 194 background), the quantification of the volumes is automatic by the Avizo 195 Software. To quantify the volume of pores, and the volume of the matrix, 196 the software uses the voxel size of the image, the 3-D reconstruction, and the 197 segmentation. The technical features of MicroCT Scanner images include 198 resolution (voxel size) between 0.0265 and 0.0443mm, detector 2000×2000 199 pixels with each pixel spaced at $200\mu m$, 2500 projections, angle steps of 200 0.144° , and X-Ray energy of 190kV. 201

202 3.4. Plasticity Index

The plasticity index has been obtained for the studied samples. For each sample, we have performed three tests for reliable results. First, a Plastic Limit test was performed, and second a Liquid Limit test using Casagrande's liquid limit device. Both tests, liquid limit and plastic limit, have been performed following the standard approach in soil mechanics (Das, 1941). The geological material used for the tests was the powder size of the core samples (μm) mixed with small flakes (mm).

²¹⁰ 4. Meso-scale characterization of the shearing zone material

In this section, we explain the methodology followed to carry out a proper meso-scale characterization of the core samples to test in the thermal triaxial apparatus. Firstly, we define the protocol performed to remold the core samples from the S10 borehole. Then, we explain the experimental protocol we have carried out to obtain the optimal confining pressure to perform the thermal and rate tests, the critical-state line, cohesion, and the angle of friction coefficient of the material (black shales of the Silurian).

218 4.1. Remolding of the samples

The core samples from the landslide were in a dry state due to their 219 preservation (Fig. 1c). Therefore, they have been healing in a humidity 220 chamber for three months at 85% humidity. The sample's grain size has 221 been defined by three main sizes: chips from dm to cm, flakes from cm to 222 mm, and powder size as μm . The material chosen to test in the triaxial has 223 been the powder size mixed with mm flakes (Fig. 2a). The samples were 224 mixed with water (Fig. 2b) to reach an average humidity of 70% at a room 225 humidity of 45% and a room temperature of $20^{\circ}C$. The saturated material 226 was placed in cylindrical molds of 35mm in diameter and 85mm height by 227 applying compaction to reduce voids once the sample dried. The molds with 228 the soil have been drying for 48 hours in the oven at a temperature of $50^{\circ}C$. 229 Afterward, they have been 6 hours at room temperature to cool down and 230 removed the molds to let the sample reach room temperature and dry for 24 231 hours before cutting and preserving them, covered in a plastic bag, in the 232 fridge until their test. The samples tested in the thermal triaxial machine 233 have a size of 35mm in diameter and a height between 65 - 70mm height 234 (Fig. 2b), with an average sample temperature of $20^{\circ}C$ and 40% humidity, 235 and an average room temperature of $21^{\circ}C$ and 50% humidity. 236

The remolded samples have been tested in a customized thermal triaxial machine (Fig. 2d,e,f) to characterize the thermal and rate sensitivities of the Silurian shales of the inside and outside the shear band. The core samples have been tested with the same characterization (i.e., every half a meter
depth) as the micro-scale tests.

242 4.2. Experimental protocol

For the tests performed on all the samples in the thermal triaxial ma-243 chine, we have always followed the same steps in the same order to have 244 consistency in the results and repeatability. The cylindrical samples, 38mm 245 diameter and 65 - 70mm height (Fig. 2c), are not consolidated after being 246 remolded at the reported field humidity. Therefore, we have first performed a 247 triaxial compression on the samples outside the shear band at varying pres-248 sures between 50 - 700 kPa in undrained conditions (Fig. 3a). With the 249 data of the tests performed, we can calculate the mean effective stress as 250 $p' = \frac{\sigma_1}{3} + 2\frac{\sigma_3}{3}$, being $\sigma_1 = \frac{LoadCell}{SampleArea}$, and σ_3 the confinement pressure. We 251 also calculate the corrected deviatoric stress as $q = \sigma_1 - \sigma_3$. To obtain the 252 critical state line of the material (Fig.3b), we plot the calculated values of 253 p' and q of each confinement pressure and calculate the linear interpolation 254 between the points. The equation of the critical state line is presented in 255 Fig.3b. The slope, M_{cs} , of the critical state line is 1.3, and the friction angle 256 of the material at critical state is calculated as $M_{cs} = \frac{q}{p'} = \frac{6sin\phi_{cs}}{3-sinn\phi_{cs}}$, and has 257 a value of 32.52° , and a cohesion of 213.3kPa (Santamarina & Cho, 2001). 258

Once the material has been characterized, we have tested all the samples (outside and inside the shear band) to determine their rate and thermal sensitivities. As the material was limited, due to the samples being from the S10 borehole (Fig. 1c) and only being able to remold them twice, we have performed thermal and rate sensitivity tests at a critical state in each test as follows:



Figure 2: Remolding of the samples and the customized thermal triaxial machine. (a) Dry state of the material of the S10 borehole after being in the humidity chamber. The size of the material is powder size mixed with *mm* flakes. (b) Material of Fig. 2a saturated in water with an approximate humidity of 70%. (c) The final shape of the remolded sample (Fig. 2a,b) after being dried in the oven and cut. This figure represents the samples tested in the customized thermal triaxial machine. (d) The base of the cell, the triaxial frame, and the three pumps. (e) The thermal cell in the triaxial frame, where the orange pads are the heating sources to increase the temperature in the cell and the sample. (f) Thermal triaxial machine with the protector case for the thermal tests and the sensors controller for the piston and temperature on the right.



Figure 3: Meso-scale tests to characterize the critical state line of the material. (a) Axial strain [%] with deviatoric stress [kPa] of the triaxial tests performed on the landslide material at different confinement pressures. (b) Mean effective stress (p') [kPa] with deviatoric stress (q) [kPa] of each confinement pressure of Fig. 3a. The graph also shows the stress paths of each p' - q point, the equation of the critical state line, the friction angle at the critical state (ϕ') , and the value of cohesion of the material (c').

First, a triaxial compression at the confinement of 200kPa. When the ra-265 dial compression has reached the value of 200kPa and holds the pressure, we 266 have started the axial load with a velocity of 0.5mm/min with two loading-267 unloading cycles at 1 and 5% of axial strain (Fig. 4a). These two cycles of 268 loading-unloading were performed to eliminate any inertia effects stemming 269 from the frame's rigidity. We let the axial load continue, at a constant axial 270 rate and constant confining pressure, until the sample reaches a critical state 271 at which the deviatoric (differential) stress (q), confining stress, volume, pore 272 pressure, and temperature remain constant (Fig. 4a). After these two cycles, 273 the sample has let to reach a critical state until approximately 12% of axial 274 strain, and the velocity stepping test starts. While at a critical state, velocity 275 stepping is performed by increasing the axial load at five constant rates (i.e., 276 velocities) from 0.0001 - 1mm/min, allowing the sample to relax to a new 277 critical state before performing the next velocity step (Fig. 4b). Through 278 this exercise, the rate sensitivity of the material's shearing resistance at a 279 critical state, q_{cs} , is evaluated to be: 280

$$q_{cs} = q_{ref} \left(\frac{V}{V_0}\right)^N \tag{1}$$

where V is the velocity [mm/min], N is the rate sensitivity of the material [-], q_{ref} and V_0 are the reference values for the shear resistance (deviatoric stress at critical state [kPa]) and the velocity [mm/min], respectively. For the Silurian shales outside and inside the shear band, the parameters obtained from this part of the tests in all the samples are: N = 0.0136, $V_0 = 1mm/min$ and $q_{ref} = 719.4kPa$.

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Once the velocity steps finish, the sample is kept at a critical state by

holding its volume and allowing it to relax to the slowest critical state pos-288 sible by the machine's specifications, at $V = 10^{-9} mm/min$. At this point, 289 the thermal tests start (Fig. 4a) by keeping the confining pressure and load-290 ing velocity constant and only increasing the temperature slowly at steps of 291 $2 - 3^{\circ}C$, inducing a $1.5^{\circ}c$ per hour rate, before letting it equilibrate to a 292 new critical state for a couple of hours (Fig. 4c). The temperature of the 293 sample was monitored with a thermal probe less than 10mm away from the 294 sample, and the temperature was held constant until this probe stabilized to 295 a steady state. Once the temperature of the sample and the axial stress are 296 equilibrated, at each temperature variation, we mark the deviatoric stress 297 values to obtain the thermal sensitivity of the material defined as: 298

$$q_{cs} = q_{ref} \exp\left(-M\Delta T\right) \tag{2}$$

where $\Delta T = T - T_{lab}$ is the temperature variation from the base value of $T_{lab} = 20^{\circ}C$ and M is the thermal sensitivity coefficient [° C^{-1}]. For these tests we obtain $M = 0.04^{\circ}C^{-1}$ and $q_{ref} = 719.4kPa$ for the material located at the center of the shear band. While at the edges of the shear band we have obtained $M = 0.023^{\circ}C^{-1}$ and $q_{ref} = 695.15kPa$, and outside the shear band $M = 0.014^{\circ}C^{-1}$ and $q_{ref} = 704.19kPa$.

305 5. Results

In this section, we show the results of the micro-and meso-scale tests performed on the core samples of the shear band of the Cal Ponet - Cal Borronet lobe. The micro-scale tests performed have also been previously presented by the authors (Seguí *et al.*, 2021). The meso-scale tests are presented below



Figure 4: Experimental tests. (a) Graph of axial strain [%] and deviatoric stress, q [kPa], showing the evolution of the axial load in isotropic compression with two axial load-unload cycles, velocity steps, and increase of temperature. The inlet is an SEM-EDS image of the shear band sample, showing the orientation of the phyllosilicates at the center of the shear band (muscovite [grey], chlorite [white], and paragonite [dark grey]) parallel to the shearing direction (Seguí *et al.*, 2021). (b) Graph of time [min] with different axial rates [mm/min] applied to all the tests performed. (c) Graph of time [s] and the temperature of the sample [°C] with an average increase of temperature of $1.5^{\circ}C/hour$.

310 in detail, but part of them have been presented previously by the authors

- ³¹¹ (Seguí & Veveakis, 2021).
- 312 5.1. Micro-scale tests

313 5.1.1. XRPD

A total of six samples have been tested in this study from the S10 borehole. From the XRPD tests, 5 mineral phases have been found: quartz $[SiO_2]$, muscovite $[K_2Al_4Si_6Al_2O_{20}(OH, F)_4]$, chlorite chamosite $[(Mg, Al, Fe)_{12}(Si, Al)_8O_{20}(OH)_{16}]$, paragonite $[(K, Na)_2Al_4Si_6Al_2O_{20}(OH, F)_4]$, and calcite $[CaCO_3]$.

Fig. 5 presents the results of the quantitative analyses (in wt%) of the 318 mineral phases present in the six samples studied. The sample 27 - 27.5m is 319 formed by quartz (35wt%), muscovite (52wt%), chlorite chamosite (13wt%), 320 and with minor presence of calcite ($\leq 1wt\%$). The rest of the samples 321 analyzed by XRPD, present a very similar composition and proportion of 322 the identified minerals, which are: 27.5 - 28m, 28 - 28.5m, 28.5 - 29m, 323 29 - 29.5m, and 29.5 - 30m. In all the analyzed samples, the presence of 324 quartz varies between 24 and 21wt%, muscovite between 59 and 53wt%, 325 paragonite between 22 and 14wt%, and chlorite chamosite between 6 and 326 3%. 327

Therefore, the changes in the colors of the core samples seen in Fig. 1C are explained by the mineralogy. The sample 27 - 27.5m, with high content in chlorite, presents a green tone, and samples from 28 to 30m depth have a high content in muscovite and paragonite which makes the material look darker (black-grey tones also influenced by the organic matter).

333 5.1.2. SEM-EDS

SEM-EDS analyses have confirmed the mineral composition of the phases found in the samples analyzed by XRPD. In the case of paragonite, EDS analyses reveal the presence of Na in the chemical composition. Moreover, phyllosilicate rich in potassium found is characterized as muscovite due to the amount of K in the mineral composition (Deer *et al.*, 1962). The samples analyzed from the S10 borehole represent the shear band and its surroundings showing four different textures as follows:

- (i) Sample 27 27.5m. This material is a black-gray-green shale with
 several small fractures filled by quartz (Seguí *et al.*, 2021, Fig. 6)
 surrounded by phyllosilicates (muscovite, paragonite, and chlorite).
 Muscovite particles are randomly oriented (Fig. 5) but show a fluidal
 texture of the platy grains of muscovite, with face-edge and face-face
 contacts. Quartz grains are found inside the sub-horizontal fractures
 with different thickness sizes between bands of phyllosilicates.
- (ii) Sample 27.5 28m. The texture of this sample is different from the 348 rest. SEM-EDS images show large fragments of black-grey-green shales 349 (Seguí et al., 2021, Fig. 6) and, between them, small particles of quartz 350 and phyllosilicates (muscovite, paragonite, and chlorite) with face-face 351 and face-edge contacts. The texture inside the large clasts is identical 352 to the texture of the 27 - 27.5m sample. These large fragments are not 353 matrix-supported as the small particles of quartz and phyllosilicates 354 are not attached (Fig. 5a,b). 355
- (iii) Samples 28 28.5m, 28.5 29m, and 29.5 30m. These samples are black-gray shale with small $(14\mu m)$ and large (over $75\mu m$) fractures

(filled by quartz), located between layers of phyllosilicates (muscovite, 358 paragonite, and chlorite) and arranged forming folds. Phyllosilicate 359 particles have a certain orientation and direction, following the shape 360 of the folds (Fig. 5c,d), and their contacts are mainly face-edge, but 361 with some face-face contacts. Quartz grains are located between phyl-362 losilicates following the orientation of the folds (Seguí *et al.*, 2021, Fig. 363 6). In these samples, muscovite particles and chlorite-muscovite are 364 present as intergrowths (Seguí et al., 2021, Fig. 6). 365

(iv) Sample 29 - 29.5m is where has been found the center of the shear 366 band. It is a homogeneous and dark sample with large and oriented 367 fractures filled with quartz. SEM-EDS images show a well-defined ori-368 entation and direction of the phyllosilicates -muscovite, paragonite, and 369 chlorite- (Fig. 5e,f, and (Seguí et al., 2021, Fig. 6)). This sample also 370 presents thick fractures where, at their boundaries, the phyllosilicates 371 are aligned (by their faces) parallel to the fractures (Seguí *et al.*, 2021, 372 Fig. 6). Furthermore, contacts between crystals of phyllosilicates are 373 face-face (Seguí et al., 2021, Fig. 6). 374

The sizes of the phyllosilicates, in the analyzed samples, have been obtained by SEM-EDS, and vary between 6 and $50\mu m$.

377 5.1.3. MicroCT

Six samples of the S10 borehole have been tested to study the porosity of the material by MicroCT Scanning. The results are shown in Fig. 5, which presents the percentage of porosity of each sample, allow us to see the evolution of the porosity along with the shear band as follows:

- (i) Samples 27-27.5m, 27.5-28m, and 28-28.5m. These samples present a matrix volume fraction of 0.226, 0.2, and 0.23 and a porosity volume fraction of 0.0029, 0.0027, and 0.0027, respectively. Thus, the samples have a percentage of pores of 1.27%, 1.37%, and 1.15%, respectively (Fig. 5). The porosity in these samples have a defined orientation and direction of small pores as well as some large fractures (Seguí *et al.*, 2021, Fig. 7).
- (ii) Samples 28.5 29m and 29.5 30m. These two samples have a matrix 389 volume fraction of 0.14 and 0.22, and a porosity volume fraction of 390 0.00021 and 0.00019. Thus, with a percentage of pores in the sample 391 of 0.14% and 0.087%, respectively (Fig. 5). The porosity in these 392 samples is shown as very thin and large fractures, with an NW-SE 393 direction (of the image (Seguí *et al.*, 2021, Fig. 7), not the field), 394 across the entire sample. Furthermore, the samples present small pores 395 aligned and organized in bands, in the same direction and they look 396 like thin fractures. 397
- (iii) Sample 29 29.5m. This sample presents a matrix volume fraction of 398 0.14 and a porosity volume fraction of 0.0033. Thus, with a percentage 399 of pores in the sample of 2.16% (Fig. 5). This sample presents, at its 400 center, a large fracture (Seguí et al., 2021, Fig. 7) with large intercon-401 nected pores (where fluid can flow through them). This large fracture 402 has an E-W direction (of the image (Seguí *et al.*, 2021, Fig. 7), not the 403 field). Above and below this fracture, there are thin fractures (some of 404 them located at the bottom of the sample with larger pores) oriented 405 parallel to the shear movement. 406

407 5.1.4. Plasticity Index

The plasticity index results are presented in relative values (i.e., all re-408 sults are divided by the value obtained for the first sample, 27 - 27.5m). 409 We present the results in relative values because 1) the values obtained in 410 Liquid Limit and Plastic Limit tests usually depend on the user, as each user 411 adds a different amount of water to the samples; and 2) when the borehole 412 was performed, the company obtained a plasticity index value of 8.7 for the 413 sample 27.15 - 27.30m and now we obtained a plasticity value of 3.96 for the 414 sample 27 - 27.5m. This difference in the value of the plasticity index could 415 also be due to the current dry state of the samples and their preservation 416 after 12 years (since the borehole was performed). However, as the sam-417 ples were uniformly exposed to the environment, we assume they degraded 418 proportionally. 419

The results of the plasticity index reveal the same behavior as the porosity 420 results (Fig. 5). These results show three distinct values that can be identi-421 fied as three groups (same groups as MicroCT). Firstly, samples 27 - 27.5m, 422 27.5 - 28m, and 28 - 28.5m, with values of relative plasticity index of 1, 0.93, 423 and 0.97, respectively (Fig. 5). Secondly, samples 28.5-29m and 29.5-30m, 424 with relative plasticity values of 0.67 and 0.62, respectively (Fig. 5). The 425 samples of the second group have lower values of relative plasticity index 426 compared to the values of the first group (over 30% lower). And, thirdly, 427 sample 29 - 29.5m, has a value of relative plasticity index of 1.95 (Fig. 5), 428 being a higher value of relative plasticity index compared to the rest of the 429 samples. 430



Figure 5: Results of the micro-scale tests. Mineral phases found by XRPD at each depth in *wt*%, the porosity of each sample in %, and the relative plasticity index for each sample. Displacement data from the inclinometer (Fig. 1b, (EuroconsultSA, 2017)) is shown to correlate the results of the tests performed with the vertical deformation in the field. (a,b) SEM-EDS images of 27.5-28m sample, showing the randomly oriented and grain supported texture. (c,d) SEM-EDS images of 28.5-29m sample, showing the reorientation of the minerals forming smooth folds. (e,f) SEM-EDS images of 29-29.5m sample, showing the alignment of the minerals parallel to the shearing direction.

431 5.2. Meso-scale tests

The results obtained in this study, performed in the thermal triaxial with velocity stepping and temperature increase, are presented in Fig. 6a. The rate sensitivity for all the samples tested is the same for the Silurian shales of the S10 borehole of the Cal Ponet-Cal Borronet lobe. The data points of the rate sensitivity (axial rate - deviatoric stress at critical state) obtained for all the samples follows a power law. The equation of rate sensitivity obtained in this study is the following:

$$q_{cs} = 719.4 \cdot V^{0.0136} \tag{3}$$

where the value of rate sensitivity obtained for all the samples is N = 0.0136, for reference deviatoric stress of q = 719.4kPa and reference velocity $V_0 = 1mm/min$.

Moreover, the data points for the three thermal sensitivities (outside, inside, and at the edges of the shear band) are presented in Fig. 6b. The tests on each sample have been carried out twice, as it was the maximum times that the material was able to be remolded, allowing thus to obtain more data points to determine with accuracy each equation law, which is the following:

$$q_{cs} = 704.19 \cdot e^{(-0.0136 \cdot \Delta T)} \tag{4}$$

448

$$q_{cs} = 695.15 \cdot e^{(-0.023 \cdot \Delta T)} \tag{5}$$

$$q_{cs} = 719.4 \cdot e^{(-0.04 \cdot \Delta T)} \tag{6}$$

where Eq. 4 refers to the thermal sensitivity outside the shear band with a value of M = 0.0136, Eq. 5 at the edges of the shear band with a value



Figure 6: Results of the meso-scale tests. (a) Rate sensitivity of all the samples tested (outside, at the edges, and inside the shear band). The results obtained show the power law that represents the rate hardening behavior of the Silurian black shales. (b) Thermal sensitivities of outside (red), at the edges (blue), and inside (black) the shear band. The results follow exponential laws for each dataset that show the thermal softening behavior of the Silurian black shales within the area of the shear band.

of M = 0.023, and Eq. 6 at the center of the shear band with a value of M = 0.04, being the higher value of thermal sensitivity among all the samples tested.

455 6. Discussion

In this section, we discuss the validity of the experimental protocol performed on the thermal triaxial tests compared to other studies previously performed by other authors in clayey materials. Moreover, we interpret the results obtained at the meso-scale of thermal and rate sensitivities related to the fabric (orientation of the minerals) and the mineralogy of the samples tested at the micro-scale.

462 6.1. Validity of the experimental protocol

As we have mentioned previously in the Introduction section, Vardoulakis 463 (2002b) presented a mathematical model for the thermal and rate behavior 464 of clayey shear bands, that accounts for thermal softening and rate hardening 465 of the material when shearing. In this mathematical model the author cou-466 ples the thermo- and visco-plastic behavior of the clays. In that paper, the 467 laboratory results presented were from Leinenkugel (1976), who performed 468 tests on remolded kaolin clay to obtain the strain-rate sensitivity, and Hicher 469 (1974) that performed tests on black clay (kaolinite) to obtain the thermal 470 friction sensitivity of the material. Leinenkugel (1976) obtained the power 471 law of Eq. (1) to describe the shear stress behavior with the strain-rate and 472 the rate sensitivity of the material, which is a rate hardening law (i.e., when 473 increasing the velocity/strain-rate, the shear stress increases). Leinenkugel 474 (1976) obtained a value of rate sensitivity for kaolin of N = 0.01 (Table 1). 475 Rate sensitivity data from other authors is also presented in Fig. 7a, show-476 ing the same power law and rate hardening, the same as the one obtained 477 in this study. Samuelson & Spiers (2012) performed direct shear with veloc-478 ity stepping in fault gouges material formed by high content on illite (Fig. 479 7a), with a value of rate sensitivity of N = 0.0079 (Table 1). Ikari *et al.* 480 (2009) performed velocity stepping tests in a biaxial stressing apparatus on 481 a synthetic montmorillonite-rich gouge (Fig. 7a), obtaining a value of rate 482 sensitivity of N = 0.0116 (Table 1). 483

Regarding thermal sensitivity, Hicher (1974) performed triaxial tests to see the evolution of the material with temperature. The latter obtained a thermal softening behavior of the clayey material (i.e., when the temperature

increases, the residual friction coefficient of the material decreases), with the 487 result of the exponential law of Eq. (2). This exponential law defines the 488 residual friction coefficient as the reference friction coefficient by the expo-489 nential thermal sensitivity of the material and the difference between actual 490 and reference temperatures. Hicher (1974) obtained a value of thermal sensi-491 tivity for kaolinite of $M = 0.0093^{\circ}C^{-1}$ (Table 1). Additional data of thermal 492 sensitivity from other authors is shown in Fig. 7b, where the law is the 493 same exponential law as Hicher (1974) obtained, and is the same as the one 494 obtained in this study, showing a thermal softening of the clayey materials. 495 Robinet, J.-C. et al. (1997) performed thermal test in a oedometer on kaoli-496 nite (Fig. 7b), with a thermal sensitivity value of $M = 0.021^{\circ}C^{-1}$ (Table 497 1). Hueckel et al. (2009) performed temperature increase in triaxial tests on 498 carbonate clays (Hueckel et al., 1998), with a value of thermal sensitivity of 499 $M = 0.003^{\circ}C^{-1}$ (Fig. 7b, Table1). 500

As shown in Fig. 7, the results of the tests performed in the customized 501 thermal triaxial for the Silurian black shales of the Cal Ponet-Cal Borronet 502 lobe, allowed us to obtain the same laws for thermal and rate sensitivities 503 as the aforementioned authors for different types of clayey materials. The 504 values of thermal and rate sensitivities are within the range of the ones ob-505 tained from the other experiments presented in Fig. 7. Thus, by performing 506 both types of tests (velocity stepping and temperature increase) on the same 507 sample, due to the limitation of core material, we have been able to obtain 508 accurate values. 509



Figure 7: Validity of the meso-scale tests. (a) Comparison of rate sensitivity values obtained in this study by velocity stepping with other velocity stepping tests performed on clayey materials by other authors. Showing the values of rate sensitivity for each test and the rate hardening results. (b) Comparison of thermal sensitivity values obtained in this study by temperature increase with other thermal increase tests performed on clayey materials by other authors. Showing the values of thermal sensitivity for each test and the thermal softening results.

Table 1: Values of rate and thermal sensitivities. Reference deviatoric stress at critical state $q_{cs}ref$, thermal sensitivity M, and rate sensitivity N. The values of this table are the ones obtained from the rate and thermal laws shown in Fig. 7.

q _{cs} ref [kPa]	M [°C⁻¹]	N [-]	Material
719.4		0.0136	Silurian shales
719.35		0.0079	Illite (after Samuelson & Spiers 2012)
691.88		0.0095	Kaolin clay (after Leinenkugel, 1976)
719.54		0.0116	Montmorillonite (after Ikari et al., 2009)
719.4	0.04		Silurian shales (shear band)
704.19	0.014		Silurian shales (outside the shear band)
695.15	0.023		Silurian shales (edges of the shear band)
719.4	0.0093		Kaolinite (after Hicher, 1974)
719.4	0.021		Kaolinite (after Robin et al., 1998)
719.4	0.003		Carbonate clay (after Hueckel et al., 2009)

6.2. Relationship between mineralogy and fabric with rate and thermal sensitivity

The results of the meso-scale tests have revealed the same rate sensitiv-512 ity value of the material for all the samples tested. These results could be 513 explained by the six samples having the same mineralogy, despite their vari-514 ations in wt% of each mineral phase (Fig. 8). All the samples are, overall, 515 composed of the same three phyllosilicates (chlorite, muscovite, and parago-516 nite) and quartz. It can be seen, in Fig. 8, that the variation of the amount of 517 quartz vs. phyllosilicates does not affect the rate sensitivity of the material. 518 By comparing the values of the rate sensitivity of the Silurian black shales 519 with the other clayey materials in Fig. 7a can be seen that most of them 520 have very similar values, but their difference could be due to their content in 521 additional mineral phases and higher difference in wt%. 522

Thermal sensitivity results show different values for different depths of 523 the samples tested (Fig. 6b). This difference could indicate that it is due 524 to the effect of the fabric of each sample. The orientation of the mineral-525 ogy with face-face contacts shows a higher value of thermal sensitivity of 526 the material. Whereas, when the material is randomly oriented with face-527 edge and/or edge-edge contacts, the thermal sensitivity is the lowest one. 528 The results also show an intermediate value of thermal sensitivity for the 529 samples right above and below the center of the shear band. This seems to 530 be due to an intermediate rearrangement of the minerals (as they are ori-531 ented in smoother folds). Fig. 8 correlates the effect of the fabric in terms 532 of porosity and plasticity index with the thermal sensitivity of each sample, 533 showing almost the same behavior between them. This correlation of the fab-534

ric with porosity is because the porosity outside the shear band is randomly 535 oriented with an average value of % of pores, at the edges of the shear band 536 the material becomes more oriented with smoother folds that induce lower 537 porosity and smaller pores due to the start of this rearrangement in the form 538 of smooth folds, and at the center of the shear band the full alignment of the 539 phyllosilicates parallel to the shearing direction rearranges mechanically the 540 pores by creating a path of connected pores with the highest % of porosity. 541 Moreover, we can also correlate the fabric of the samples with the plasticity 542 index due to the contact edge-face and/or edge-edge are the stronger ones 543 that make the material less susceptible to friction (i.e., less plastic -a lower 544 value of plasticity index-), whereas the contacts face-face that we found at 545 the center of the shear band are the weakest contacts, thus, facilitating the 546 friction (i.e., more plastic - higher plasticity index). Also, the plasticity index 547 is correlated with the porosity, as outside the shear band we have a higher 548 value of plasticity index due to higher pores in the sample (i.e., allowing 540 the material to absorb more water), while at the edges of the shear band 550 and regardless of the rearrangement of the mineralogy in smoother folds, the 551 material has lower porosity with smaller pores, which causes the material to 552 absorb less water, hence being less plastic. Finally, at the center of the shear 553 band, the minerals are completely aligned but also we found higher porosity 554 with pores interconnected creating a channel that allows us to absorb more 555 water. 556



Figure 8: Correlation of the micro-and meso-scale tests. Evolution in depth of the % wt of quartz and phyllosilicates with values of rate sensitivity of each sample. Also, the table correlates in depth the evolution of % of porosity and plasticity index with the thermal sensitivity.

557 7. Conclusions

This paper presents a micro-and meso-scale study of the Silurian black 558 shales of the shear band area of a deep-seated landslide, the Cal Ponet-Cal 559 Borronet lobe inside the El Forn landslide in Andorra. The results of the 560 micro-scale tests (XRPD, SEM-EDS, MicroCT, and plasticity index) show 561 that, while the mineral phases do not change from outside to inside the 562 shear band, the fabric (orientation of the minerals) does by being randomly 563 oriented outside to completely aligned parallel to the shearing direction in 564 the shear band. This reorganization of the mineralogy affects the porosity, 565 from being non-connected outside the shear band to interconnected forming 566 a layer of porosity inside the shear band. The effect of the alignment of 567 the minerals with face-face contacts also increases the plasticity index of the 568 material. 569

Moreover, the meso-scale results of the experimental tests performed in the thermal triaxial machine show that the Silurian black shales have the same rate sensitivity, being the material rate hardening, regardless of their location (inside or outside the shear band). However, the thermal sensitivity results show different values, depending on the location of the sample (outside, inside, or at the edges of the shear band), with thermal softening in all of them.

All these results have allowed us to associate the rate and thermal behavior of the material with the mineralogy and the fabric of the samples. The rate sensitivity is directly dependent on the mineral phases present in the material, while the thermal sensitivity of the material is correlated with the fabric of the sample, by being more thermal sensitive when the minerals are completely aligned in one direction and with face-face contacts, while less
thermal sensitive when the minerals are randomly oriented with face-edge or
edge-edge contacts.

Furthermore, the newly experimental protocol followed in the present 585 study has shown that both, the thermal and rate tests, can be performed on 586 a single sample. This allows to optimize the amount of material and obtain 587 more data, especially when there is limited material from a borehole. We 588 have also validated the results of this experimental protocol by comparing it 589 to previous studies that have performed velocity stepping tests and temper-590 ature increase tests, separately, on clayey materials. Showing that the values 591 obtained in the present study are very similar to the other experimental tests 592 from several authors. 593

This paper, thus, shows the importance of the thermal and rate sensitivity of the material in the shear band of a deep-seated landslide, regarding the fabric and mineralogy, respectively. These parameters are important to consider when studying shear zones and the stability of natural hazards and how the thermal and rate sensitivities evolve when the material experiences friction/shearing movements.

600 Conflict of interest

⁶⁰¹ The authors declare that they have no conflict of interest.

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XRPD and SEM-EDS (http://www.ccit.ub.edu/ES/tm02.html) analysis 603 were made and performed at the Scientific and Technological Centers of the 604 University of Barcelona (CCiTUB). Thin sections were made at the "Servei 605 de Làmina prima" from the Faculty of Earth Sciences at Barcelona Univer-606 sity. MicroCT tests were carried out at the Duke University Shared Ma-607 terials Instrumentation Facility (SMIF), a member of the North Carolina 608 Research Triangle Nanotechnology Network (RTNN), which is supported by 609 the National Science Foundation (Grant ECCS-1542015) as part of the Na-610 tional Nanotechnology Coordinated Infrastructure (NNCI). Liquid and plas-611 tic limit tests were carried out at the Soil Mechanics laboratory at Duke 612 The authors acknowledge the help of the laboratory techni-University. 613 cian Michael Blagg in performing the plasticity index tests. The rate and 614 thermal triaxial tests were carried out at the Multiphysics Geomechanics 615 Laboratory at Duke University. Support by the NSF CMMI-2006150 and 616 CMMI-2042325 projects, and PID2019-105625RB-C21 funded by MCIN/ 617 AEI/10.13039/501100011033/ are also acknowledged. 618

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