Solid Earth Discuss., referee comment RC1
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Comment on se-2021-39

Anonymous Referee #1

Referee comment on "Micromechanisms leading to shear failure of Opalinus Clay in a triaxial test: a high-resolution BIB–SEM study" by Lisa Winhausen et al., Solid Earth Discuss., https://doi.org/10.5194/se-2021-39-RC1, 2021

General comments - overall quality of the preprint

This paper describes a high resolution microstructural study of the fine-scale processes underlying shear failure of claystones, specifically the Opalinus Clay (Shaly facies). The microstructural (BIB-SEM) work is beautifully executed and presented in terms of micrographs. The study forms a valuable contribution in that very little has been done to date on the micro-mechanisms that control failure of such materials. Moreover, what has been done previously mainly addresses samples deformed to large strains beyond failure, rather than the failure process itself. The present work focuses specifically on the microstructures and micro-mechanisms associated with initiation of shear failure and shear localization at low strains up to the peak stress, thus providing fundamental insight into and a physical basis for the formulation and testing of quantitative micromechanical models in future. Given the importance of understanding clay and claystone failure and associated permeability changes in the context of geological storage systems, the present work adds a useful observational dimension to purely continuum approaches to shear failure and localization in these materials. It is nice work, it is well presented, well illustrated and mostly well written. It certainly deserves to be published. At the same time, however, there are a substantial number of scientific and technical issues that I feel the authors need to address before the pre-print would be acceptable for full publication. These are mostly minor but quite numerous. For this reason, I recommend major revisions.

SCIENTIFIC COMMENTS

General scientific points that require attention are as follows:

A) The authors often describe their work as addressing the microphysics or micromechanics of shear failure of the OPA material. However, there is no physics or

mechanics in the paper at all – it is purely qualitative and conceptual. Rather, the authors address the MICRO-MECHANISMS involved in deformation and failure of the material studied and should really use this term to describe their work (it is a micro-mechanistic study).

B) The experimental sample investigated was tested unconsolidated and undrained at 4 MPa confining pressure. There is no discussion as to how these test conditions (hence observed processes) might relate to conditions of interest in the subsurface, such as caprock or repository conditions. Perhaps the effective stress would be comparable (low) due to consolidation effects, but some discussion of this would be welcome – as would the likely pore pressure evolution in the experiment (perhaps based on volumetric strain data).

C) The description of the experiment does not seem quite right in terms of boundary conditions applied to the sample. The authors seem to be saying that the test was carried out at constant confining pressure and at controlled circumferential strain rate, which is equivalent to controlled radial strain rate. Do the authors mean that axial loading system was servocontrolled to provide a constant radial expansion (or contraction rate)? This seems unlikely to me. Or do they mean that a constant axial strain rate (or stress rate) was imposed and the radial expansion/contraction was simply measured (not controlled)?

D) The terminology used to describe the various cracks, fractures and shear bands at different scales is somewhat confusing to the reader (at least this one!) and not very systematic. I would strongly recommend defining the terminology to be used for these features at an early stage in the manuscript. It would be useful if the authors could define cracks versus microcracks, as well as defining crack character – i.e. whether individual cracks are Mode I (opening) or Mode II (shear) cracks or mixed mode. The term "shear band" should also be defined and appropriately distinguished from a shear crack – at least in a morphological sense. Voids that open between grains due to local dilatation should also be defined as something like dilatational or dilated pores. Of course, the observed features may ALL have been influenced by unloading (removing axial stress and/or confining pressure), so that needs to be mentioned.

E) While it is clear that many of the observed cracks and shear bands are directly associated with the evolution of the macroscopic shear failure process, some features that the authors link to shear failure could potentially be present in the starting material or could have been caused by the application of confining pressure (i.e. to the hydrostatic component of consolidation). Crushed fossils, disrupted pyrite framboids, buckling of phyllosilicates etc all come to mind here. I do not doubt the authors' interpretation necessarily, but it would be desirable to add a few micrographs AT LEAST of an undeformed control sample to prove that the above features are not present in the starting material. Ideally one would like to see a few shots of a hydrostatically loaded microstructure too, if easily available.

F) Last but not least, some brief discussion is needed as to how fluid flow from consolidating regions of the sample to dilating regions of the sample may have facilitated

shear localization (as opposed to dilatancy hardening), as this could be a key component in controlling the failure behavior and strength. Such aspects would be essential for any quantitative microphysical modelling in future, and notably for understanding strain rate effects.

Detailed scientific comments/questions

1) Title: This seems unnecessarily long and not especially informative to me. Would something like "High resolution BIB-SEM study of micromechanisms leading to shear failure of Opalinus Clay in a triaxial test" not be more accurate and sufficient??

2) Abstract: Best clarify that failure occurred by shear failure and mention orientation of the shear farcture/band network (line 21).

3) Last statement about LIMITED similarity with natural fault zone microstructure and inferred role of pressure solution in nature is not very well supported in the main text. Perhaps add some additional evidence in the main text?

4) Intro: I advise using the term "radioactive waste" rather than "nuclear waste". Nuclear suggests high level waste risk which is often not the case. Atomic nuclei are also hardly waste.

5) Page 2, line 48. Microstructure is perhaps the wrong term here. Mineral composition would be accurate. Line 52 – what measure of visible pore size shows a power law distribution??? Lines 61-62 – compressive loading of samples in what orientations with respect to bedding? And what is meant by plastic flow – please define as it has many meanings.

6) Page 3, Line 83: Is "slickensides" the correct term here for these very fine striations in the mirror slip surface? Are they optically visible or only in SEM and sub-optical? Line 88 - do you mean smectite interlayer water here? Please clarify.

7) Page 4, Line 124: Mixed layer silicates?? Do you mean mixed layer smectites and smectite-illite??? Last line: what is the pore fluid? A brine with what composition?

8) When first describing fracture/crack/bedding/loading orientation, please define what you mean by orientation or inclination in terms of an angle (say theta) defined in a small diagram. Present usage is confusing, perhaps due to language. It is important to be

consistent in the use of this terminology throughout the paper. Confusing at present.

9) Section 2.2. Please clarify boundary conditions imposed in the experiment – see point B above.

10) Section 2.3, first line. Mechanically stabilized with epoxy ?

11) Section 3.1, last 3 lines. Specify branching fractures as the green ones if that is what is meant (all look branching to me). Fragments are only CRUDELY lens-shaped – best say this perhaps. Please clarify what is meant by "relay fractures".

12) Section 3.2, first paragraph: How long were the samples stored before study ?? Please indicate in the material description. What reacted to produce gypsum? How do you know this occurred during sample storage?? Could storage have had any other effects that could corrupt the microstructure?

13)Page 6, Line 183: Microcracks were often oblique to bedding…. Which set is meant here?

14) At several points in the description of the microstructure (Results – Figs 4 and 5), reference is made to increased porosity and grain-matrix separation at specific sites around clasts and clast-like/sigmoidal micro-lithons. These sites seem to correspond to interfaces under local extension – i.e. at roughly 45 degrees to the overall (macroscopic) shear failure plane orientation. This should be described as such, if indeed a widespread feature, making reference to previous work that has observed and constructed models for this type of feature (e.g. Den Hartog & Spiers 2014; Haines et al., 2013). These features have key mechanical significance in defining the onset of mechanical weakening due to a decrease in dilation angle (see papers by Den Hartog et al).

15) Page 8, line 235: "Plastic reorientation of clay aggregates". What exactly is meant here? Why is this plastic not frictional intergranular sliding? "Ductile" deformation is inferred from reorientation to form an SPO, I guess? The term "plastic" is used elsewhere in the ms to refer to intracrystalline plasticity of the phyllosilicate grains> perhaps make more consistent? Line 236 – "grain boundary sliding". This term is generally used for a high T process involving grain boundary dislocations. Better use "intergranular sliding" here as it is likely a frictional or fluid-lubricated sliding process. Lines 239-242 – I am surprised that no reference is made here to the top quality work on deformation of claystones such as COX by the Paris Est (Ecole des Pont) Team (recent papers by Philippe Braun, Pierre Delage and co-workers). Relevant work should be added.

16) Page 9, Line 260: It is assumed that unloading elasticity is equal throughout the sample. Perhaps make this "roughly equal" as the elastic stiffness of the porous zones will certainly be much lower than the undeformed matrix. Line 268-269 – reference is made here to the amounts of elastic versus inelastic deformation in the sample. This could be evaluated from the elastic part of the unloading curve, perhaps. Not done? Could this be added to add a littlle more quantitative information?

17) Section 4.3 - Micro-mechanical model. This is a misnomer, I feel, as the model presented is a conceptual model containing no real micromechanics – a term that implies a quantitative approach. This header is perhaps best modified to "4.3 Micro-mechanistic model" - which is probably what is meant by the authors. Perhaps it would be wise to add to this section the caveat that the conceptual model developed is based on a low P experiment with no pre-consolidation, and to clarify the imposed boundary conditions (constant P, controlled axial or radial? strain rate etc etc). It has to be recognized that different microstructures may develop under different conditions, or it should be argued why this would not be the case.

18) Fig 9 vs. Fig 1. This depicts the micromechanistic model proposed. However, the stress strain-curves used to illustrate the various stages of evolution looks rather different from the original experimental data depicted in Fig.1. Is the stress axis perhaps the total axial stress as opposed to the differential stress plotted in Fig. 1?? Please clarify as the stress levels mentioned seem not to match up as is. It would be useful to clarify the orientation of Sigma-1 in the microstructural sketches also. In Fig. 1, how was the stress level labelled "crack initiation" established? This should be mentioned somewhere?

19) Section 5 - Implications and conclusions. As indicated above and in my general scientific comments, this section would benefit from some brief consideration of the extent to which the observed processes and proposed mechanistic model can be expected to apply under shallow upper crustal (geo-storage system) conditions. The authors do state that more work is needed on consolidated samples and on naturally sheared/faulted samples to test the broader applicability of the model. However, in future experiments, would not higher P-T conditions and deformation/loading rate not be important too, bearing in mind issues like smectite hydration state changes and internal fluid pressure equilibration, for example?

20) Still Section 5: In Line 353 of page 12, how does the present study support implementing damage into constitutive modelling in the references mentioned? Do the authors mean "support" here or "provide input to constrain" such models, perhaps?

21) Figures 2-7: It would be useful if the orientation of the principal compression direction is indicated in all key micrographs, either directly in the micrographs or appropriately via the captions. Angles defining fracture and shear band orientations should be marked on key micrographs also, so that the intended meaning of terms like inclination (to what? Horizontal? Vertical? Bedding?) is clear. What is OM in Fig. 5a?

TECHNICAL ISSUES (language, typographics etc)

Overall the paper is well written and in good English. Nonetheless a few small improvements can be made as follows:

i) Fault gouge spelling: Gouge NOT Gauge

ii) “Associated with” is the correct usage, not “associated to”

iii) “Indicated “should not be used to mean “correspond to”. Use the latter.

iv) A typo: Saddle-reef pores. Better define the term also as it is from structural geology and is not familiar in rock mechanics.

v) “Acting on”… not “acting to..”.

vi) “growed” >>>> grew

vii) Page 11 – reformulate first sentence. English awkward/unclear.

Finally, I would recommend a thorough spelling/grammar check at the end of the road.

I wish the authors good luck in revising the paper and hope that my comments are helpful and not too burdensome. Please note that I have elected not to do a second review, not out of disinterest but from "review overload".