Dear Dr Kirilova and coauthors,

I have now carefully read your revised paper and also considered James Gilgannon's review of that version of your manuscript and came to the conclusion that, in its current form, the manuscript is not yet ready to be published in SE.

I do think though that with a rework that addresses the concerns, your paper will make a robust and well-cited contribution, and I look forward to receiving your revised version.

Best regards, Florian Fusseis

Dear Dr Fusseis,

We are very grateful for your time and constructive feedback. We acknowledge your expertise in porosity analyses and CT-data interpretation. Our interest is to produce a valuable contribution to the scientific society, thus we have carefully considered all of your comments. Please find our responses in green text below.

Kind Regards,
Martina Kirilova
On behalf of co-authors:
Virginia Toy, Katrina Sauer, François Renard, Klaus Gessner, Richard Wirth, and Xianghui Xiao

\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*\*

I see a certain disconnect between the data you present and your discussion, in particular Section 5.3, in particular, it isn't obvious to me that your data really support the conclusions you draw there. This doesn't mean that the conclusions are incorrect, but rather that the trends in the data you report are not overly obvious.

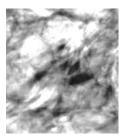
In particular, I have concerns about the validity of the shape analysis, which hinges entirely on the covariance matrix. The matrix really describes the shape of an ellipsoid, not a pore. I would argue that for the conclusions to be robust, one would need to demonstrate that the eigenvalues/vectors indeed are a good representation of pore shapes. Pores in deformed rocks can have very convoluted shapes which are difficult to describe with standard mathematical operations. There are, however, more sophisticated tools, such as Minkowski funtionals, which you could use.

#### Response:

We realize that it is possible to characterize shapes of non-ellipsoidal features using more complex methods than fitted ellipsoids. However, it is not true that the covariance matrix approximates the pores as ellipses. Instead, the covariance matrix can be calculated on any shape. For an ellipse, the 3 eigenvalues will represent exactly the 3 axis of the ellipse. For more complicated shapes, the 3 eigenvalues measure length scales of the object in 3 perpendicular directions of space. In addition, pores detected in our samples are predominantly ellipsoidal (see figure below).

Furthermore, fitting ellipsoids is a standard and widely employed method in the field of porosity analysis. For example, Menegon et al., (2015) used fitted ellipsoids to estimate the length of the longest axis of pores in monzonite ultaramylonites. The editor is a co-author of this manuscript. This means our data are easily compared to measurements made in prior studies. In addition, Sufian et al., (2018) noted that their pore orientation tensor (which is the eigenvectors of a fitted ellipsoid) is of a similar form to

the Minkowski interface tensor. Thus, we do not think that undertaking the analysis with Minkowski functionals as recommended will substantially change our results. We have not performed a new analysis.



Ellipsoidal pores in sample DFDP-1B 69 2.54 (Fig 8a in the manuscript)

My second major concern arises from the polynomial fitting - where really the R^2 values aren't too great. I suspect, on the basis of my own experience, that you could yield substantially different porosity values quite easily if you used slightly different fits.

## Response:

We acknowledge the concern that R^2 values are "not too good". The method we used is designed to estimate and exclude fractures within the sample data, which means the R^2 will be low. We can observe microfractures present even at small volumes and this method allows us to estimate when those fractures begin to adversely affect a total porosity calculation. We are not optimizing for R^2, we are biasing the fit to smaller volumes where more pores exist, and biasing the fit against higher pore volumes where more fractures exist. A consequence of this is that the R^2 values are lower, but we believe it results in a more accurate estimate of total porosity excluding fractures.

## Specific comments (line numbers)

98 I think it might be helpful if you very briefly, as obviously described in detail elsewhere, mentioned how these samples are related to the activity of the fault. A figure (BSE images and corresponding slices through uCT data) showing the microfabrics of the four samples should be added (and can replace some of Figs 1 and 2). Also, mineral compositions, grain sizes and proportions should be reported in a table. This is relevant for your section 5.2 below.

## Response:

Section 5.2 discusses our data in the context of published studies where all the information required by the editor is described in detail. However, images of the samples are available as part of an unpublished PhD thesis (it is currently in final revision stage). We could include them but would then want to add the student's name to our author list. Is that an acceptable solution?

103 Please report on the sample-detector distance, as this is important to estimate a possible phase contrast in the data.

# Response:

The sample detector distance was 70 mm. Information added in line 105.

103 The sample size is only of minor relevance here, since the field of view will only be just over 2 mm high at 10x magnification (which I believe gives 1.3 um voxel size). What is the widths of your FoV?

## Response:

The width of FOV was 2.81 mm. Information added in line 105.

# 106 Why no phase retrieval in reconstruction?

## Response:

These samples were analyzed in 2011 before such methods were implemented at APS.

111 What are the dimensions of the sub-samples, how did you choose them, and are they statistically relevant?

## Response:

The subsamples were cropped from representative areas of the whole sample that show typical mineral associations and porosity distribution within a sample. The size of the sub-samples ranges between 601^3 voxels and 801^3 voxels. Differences in the sub-sample sizes depend on the quality of the whole sample (e.g some samples were cut smaller to exclude artifacts or big fractures, which were obviously induced by coring and/or sample preparation).

113 Why use a primitive thresholding tool, where much more sophisticated tools for local crack detection are available (e.g. Voorn et al., 2013, 2015, Ma et al., 2020)? This is especially relevant since the entire paper hinges on the analysis of your segmented porosity.

Voorn, M., Exner, U., & Rath, A. (2013). Multiscale Hessian fracture filtering for the enhancement and segmentation of narrow fractures in 3D image data. Computers & geosciences, 57, 44-53.

Voorn, M., Exner, U., Barnhoorn, A., Baud, P., & Reuschlé, T. (2015). Porosity, permeability and 3D fracture network characterisation of dolomite reservoir rock samples. Journal of Petroleum Science and Engineering, 127, 270-285.

Ma, X., Kittikunakorn, N., Sorman, B., Xi, H., Chen, A., Marsh, M., ... & Skomski, D. (2020). Application of Deep Learning Convolutional Neural Networks for Internal Tablet Defect Detection: High Accuracy, Throughput, and Adaptability. Journal of Pharmaceutical Sciences.

## Response:

We are very aware of the persisting problems with crack/fracture analyses by using simple thresholding tools. However, this study does not focus on crack/fracture detection, but instead aims to exclude any cracks/fractures from the porosity analyses. In addition, pores in our samples appear as high contrast very dark grey-scale materials. In this case, the binary thresholding tool implemented by Avizo software is more than sufficient for robust porosity segmentation. Similar approaches have been previously used by multiple studies, including Menegon et al., 2015 and Gilgannon et al., 2017, where the editor is a co-author.

113 You also must have labelled the data, which degree of connectivity did you choose? This will significantly influence your pore statistics.

## Response:

We assume the editor is concerned about the connectivity typically manipulated within the morphological operation "connected components" (i.e. "face" vs "edge" connectivity between voxels). We used this operation purely for visualization purposes (lines 120- 122). This limitation of the data was not implemented at all in the methodology for total porosity estimates. Thus, it does not affect our results. Nonetheless, this information is added in line 116.

"To omit the cracks, thresholded components with volumes larger than the volume of 200 face connected voxels (439.4  $\mu$ m3) were excluded from the binary label images"

119-128 This approach could be discussed considering the findings of Keller et al., 2011, 2013a (and others) on the porosity distribution in clays (as these being related to gouges).

Keller, L. M., Holzer, L., Wepf, R., & Gasser, P. (2011). 3D geometry and topology of pore pathways in Opalinus clay: Implications for mass transport. Applied Clay Science, 52(1-2), 85-95. Keller, L. M., Holzer, L., Schuetz, P., & Gasser, P. (2013). Pore space relevant for gas permeability in Opalinus clay: Statistical analysis of homogeneity, percolation, and representative volume element. Journal of Geophysical Research: Solid Earth, 118(6), 2799-2812.

## Response:

Thank you for the relevant references. Both studies are valuable contribution to the field of porosity analyses. However, they use only SEM and FIB data and do not integrate CT-data in their studies. Consequently, their approach is very different from ours and also their analyses mostly focus on pore size distribution and pore geometry. Instead, here we focus our interpretation and conclusions mainly on total porosity estimates. In our manuscript, we propose a method for total porosity calculations that in mathematically robust approach estimates total pore volumes while excluding cracks and fractures from the overall percentage. Excluding cracks from our datasets is fundamental because we are dealing with drill-hole samples that have suffered fracturing during exhumation.

# 121 "with limit of" is misleading, is this smaller or larger than 200 voxels?

## Response:

Thank you. The text was modified to "limited up to 200 connected components"

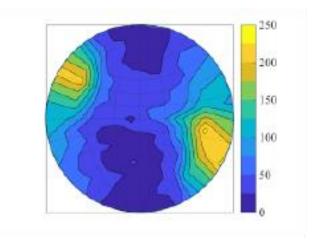
130 10 voxels as a lower limit is too small, consider the limited number of shapes you could build out of 10 cubic lego bricks - this inevitably introduces a bias in your analyses. Consider a lower limit of 5^3 voxels.

#### Response:

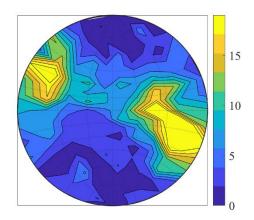
The number of shapes you can build out of 10 cubic bricks according to the sequence which defines the number of 3-dimensional polyominoes (or polycubes) with n cells is 346543, which is sufficient granularity for determining shape and orientation without bias. (N. J. A. Sloane, A Handbook of Integer Sequences, Academic Press, 1973)

In addition, introducing a lower limit of 5^3 voxels in our datasets (voxel size of 1.3 microns^3) will result in materials larger than 274.625 microns^3. Therefore, vast amount of materials of interest will be excluded. Furthermore, the shape and orientation analyses may be biased by the prevailing influence of cracks with this size.

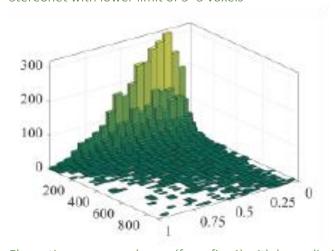
Regardless, here we present comparison between lower limits of 10 voxels and 5<sup>3</sup> voxels. We use as an example sample DFDP-1B 69\_2.54. The figures below show that a lower limit of 5<sup>3</sup> (i) does not influence the pore orientation patterns; (ii) excludes vast majority of our segmented materials which are of interest, and thus introduces bias in the resulting elongation vs pore size histogram.



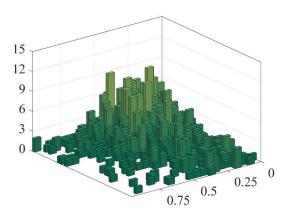
Stereonet with lower limit of 10 voxels (from figure 7)



Stereonet with lower limit of 5^3 voxels



Elongation vs pore volumes (from fig. 4) with lower limit of 10 voxels



Elongation vs pore volumes with lower limit of 5^3

135-137 I disagree with the statement that the eigenvalues describe the shape of a pore - they describe the shape of an ellipsoid, which only under fortunate circumstances will be a good approximation of the actual pore shape. To make conclusive statements on the shapes of your pores, you need to go a step further and use, e.g., Minkowski functionals (Arns et al., 2002, Lehmann et al., 2006, Wildenschild & Sheppard, 2013).

Arns, C.H., M.A. Knackstedt, and K.R. Mecke. 2002. Characterizing the morphology of disordered media. p. 37–74. K. Mecke and D.

Stoyan (ed.) In Morphology of condensed matter: Physics and geometry of spatially complex systems. Lecture Notes in Physics.

Springer, Germany.

Lehmann P et al. Tomographical imaging and mathematical description of porous media used for the prediction of fluid distribution. Vadose Zone J 2006;5(1):80.

Wildenschild, D., & Sheppard, A. P. (2013). X-ray imaging and analysis techniques for quantifying porescale structure and processes in subsurface porous medium systems. Advances in Water Resources, 51, 217-246.

## Response:

The recommended papers deal with datasets where the porosity is connected. In these studies, shape analyses using covariance matrix decomposition would not work, thus they needed to implement other techniques. However, pores in our data are separated and the covariance matrix is calculated on each single pore. To clarify this in line 133 the text was modified to:

"Individual pores in our dataset are separated (Fig. 2c), thus the covariance matrix of each pore was calculated, and the three eigenvalues of this covariance matrix were extracted."

Also, as we previously responded to the editor's major comment, the covariance matrix can be calculated on any shape. Thus, our analyses are relevant, and the use of different methods would not benefit the manuscript.

155-157 The screenshot you have added in your rebuttal to J Gilgannon indicates that your segmentation captured only a very small proportion of the actual porosity. How have you chosen the threshold value for your segmentation?

## Response:

We acknowledge that the image provided has a low resolution (due to Avizo software screenshot resolution) and may not show the threshold range properly. But we strongly disagree that we have not captured all the porosity in these samples. Pores were identified as the darkest grey-scale materials on images with good contrast, consequently thresholding was not challenging. We performed thresholding

by selecting the corresponding grey-scale range manually and individually for each sample due to slight brightness contrast differences in between samples. To ensure all pores were captured the selected threshold range was inspected throughout the 3D stack before thresholding being applied.

164-167 Fig 7 - what is the reference frame for these stereoplots? I see you are speculating that they trace a foliation, but obviously the maxima are all roughly at the same orientation between the plots.

# Response:

In these lines we do not talk about foliation. Here, we only describe the observed maximas:

"The orientations of the individual pore units show two distinctive peaks with opposite vergence, defining bipolar distributions of pore orientations"

Later in the manuscript, we only speculate that pore orientations reflect the orientation of clay minerals. The assumption is based on the predominant distribution of pores along grain boundaries of mainly clay minerals, and the abundance of intercaly pores (lines 200-206). The similar orientation of the observed orientation maxima in all samples is due to the fact that samples were drilled parallel to the foliation. This information is added in line 104.

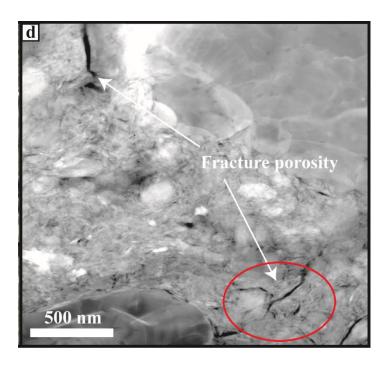
170 - "the gouges have composition..." - rephrase

Response: changed to "are composed of"

177 - What are the criteria by which you distinguish cracks from other types of pores in your TEM data? It is not really evident from Fig. 8.

## Response:

Fracture porosity was distinguished in quartz-feldspar domains (fig. 8d) where elongated, thin void spaces are associated with multiple grains and occasionally disrupt grain boundaries. The information was added to lines 178-179.



185-187 This statement is incorrect, since this is the voxel size, which does not equate to the spatial resolution.

Response: Thank you. We now say "acquisition constrains" instead of "resolution constrains"

188-190 I think this needs to be contrasted with Keller et al (2013b)'s study on Opalinus Clay. Obviously, they investigated undeformed clay as opposed to highly sheared fault gouge, but the key finding suggests that, in clay-mineral rich portions, there may be a very substantial volume of porosity hidden. What are your arguments in support of this statement?

Keller, L. M., Schuetz, P., Erni, R., Rossell, M. D., Lucas, F., Gasser, P., & Holzer, L. (2013). Characterization of multi-scale microstructural features in Opalinus Clay. Microporous and mesoporous materials, 170, 83-94.

# Response:

Thank you for this reference. We acknowledge the assumption that nano-scale porosity would not significantly influence the total porosity estimates is not robust. Thus, this statement was omitted from the manuscript.

191 (also section 3.4) How were the locations for the FIB foils chosen? How did you ensure they were representative?

## Response:

The Alpine fault samples, derived during DFDP-1, have been subjected to intense microstructural analyses both onsite and post drilling operations by the DFDP-Science team. Consequently, numerous studies have described these rocks in detail (e.g. Toy et. al., 2015; Schleicher et al., 2015). Based on these previous studies and our own observations we believe we have selected areas of interest for FIB foils that yield representative results.

192-194 I don't understand how the first half of the sentence relates to the second, and how you arrive at that conclusion.

## Response:

For better clarity the text has been modified, and now says:

"The pores visible on grain and phase boundaries in Figure 8b have similar sizes to the pores segmented on XCT images (> 1.3  $\mu$ m in diameter), thus we conclude that this is the typical habit of both nano- and micro-pores within the Alpine Fault core (Fig. 8)."

205-208 cf. Keller et al., 2013b

## Response:

We do not think that this is an appropriate reference to cite here. No change made.

209 Please restructure this section for clarity and revise paragraph structure. The reader shouldn't have to read until the end to figure out what this is about.

# Response:

We have restructured the section accordingly.

213-214 How do you identify these clay minerals as authigenic?

### Response:

We interpret these clays as authigenic because of the coexistence of newly precipitated fine materials and coarser grains, previously documented in detail by Schleicher et al., 2015.

214-216 Annotate these observations on the figure, and pls add references to support the pressure solution argument.

## Response:

The figure is now annotated. Reference added.

228 Would be good if you provided grain size distributions for the two materials, see comment for line 98.

## Response:

Grain size distribution is available as part of an unpublished PhD thesis (it is currently in final revision stage). We could include them but would then need to add the student's name to our author list. But note that substantially longer method documentation will be needed to report them here.

233 Does your analysis really demonstrate significantly different porosity values between the two cataclasite units, given the R-squared values of your regression model?

## Response:

As we previously responded to the editor's second major comment, the method used here is intentionally attempting to estimate and exclude fractures within the sample data by biasing the fit which yields lower R^2. Thus, the results presented here are robust and do demonstrated significantly different porosities. In addition, as we mention in response to reviewer James Gilgannon, similar porosity differences were also observed by calculating porosities after implementing the morphological operation "connected components".

240-251 This paragraph is a bit bold on the assumptions. First, low porosity is not necessarily consistent with low permeability, and I don't see how you verify permeability measurements? Aren't you overinterpreting a difference in porosity of 0.12%, given the uncertainties outlined in your own statistics plus the uncertainties introduced by the data processing steps and sub-sampling sites you have chosen?

## Response:

We agree that porosity is not necessarily consistent with permeability and acknowledge our bad choice of wording in this paragraph. Here, we only aim to compare our porosity data with published permeability measurements on these rocks, which show similar trend. We also indicate that our porosity estimates are in agreement with previously documented permeability gradient in these rocks. The text was modified, and we now say:

"Our data thus provide independent verification is comparable with of the permeability measurements in that study (Carpenter et al., 2014) and yields increased confidence in their interpretation of a permeability gradient with distance from the PSZ"

We strongly disagree we are overinterpreting our results, because as previously described our R2 values do not introduce uncertainties in our data (see the comment above).

254 If it is precipitation of hydrous minerals in pores (as opposed to veins), how much does the pore fluid pressure actually change as clay minerals form?

## Response:

True, some of the fluids in the pores will be included in the newly formed clays. However, because these minerals also have other components (e.g. silica), the total volume of fluid involved in their formation will not be sufficient to stop a general increase in pressure as pore size reduces. We have

added a sentence 'However this pressure increase will be slightly offset by inclusion of fluids into new hydrous minerals' (in line 263)

255 Neither Byerlee nor Sibson do actually talk about precipitation in pores (but of course about fluid pressurization), could you reformulate this slightly to avoid that impression?

## Response:

We modified the text to avoid misleading references. We now say:

"(i) very small decrease of these critically low total porosities due to mineral precipitation would cause fluid pressurization, which is a well-known fault weakening mechanism described by Byerlee, 1990 and Sibson, 1990" (now in line 263).

262 Is there graphite present in these samples? - It isn't highlighted in Fig 8. If not, please clarify this statement.

#### Response:

Graphite is not shown on the TEM images presented here. However, the presence of graphite within the Alpine Fault rocks has been previously documented and described by Kirilova et al., 2017. We modified the text to clarify this. In line 272 we now say

"which was previously documented in these rocks by previous studies (Kirilova et. al., 2017)"

Fig 2 isn't particularly helpful, since it doesn't show a workflow as such (sensu flow chart). I wonder whether 2.5D "thick slices" (see, e.g. Fig. 3 in Menegon et al., 2015, Geology) would be more better.

#### Response:

The aim of this figure is to show that the threshold range needed to segment all pores also captures cracks, and thus the later need to be removed for total porosity estimates and visualization purposes. Thus, the figure achieves the purposes it was designed for.

419 Fig caption "after the fractures" - "after removal of the fractures from the segmentated data".

Response: The text was modified.

Fig 2c, since this is a perspective figure, the scale bar does not apply to the entirety of the image, pls consider/correct.

## Response:

Thank you for this comment. We acknowledge the mistake on our side thus we removed the scale.

438/439 I don't see a difference between pores in b) and d). What are your arguments for the pores in d) being fractures?

## Response:

We acknowledge that the morphology of pores in (b) and (d) may appear to the reader as fairly comparable. However, (b) represents a phylosillicate-rich gouge area, where very elongated, extremely thin pores occur parallel to phyllosilicates orientation, thus we interpret those as "interclay porosity". Whereas in (d) pores are in association with multiple quatz-feldspar grains and occasionally affect grain boundaries. Both interclay and fracture pores with similar characteristics were observed within San Andreas fault gauges (Janssen et al., 2011)

# Micro- and nano-porosity of the active Alpine Fault zone, New

# 2 Zealand

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## 20 Abstract

- 21 Porosity reduction in rocks from a fault core can cause fluid overpressureelevated pore fluid pressures, and
- 22 consequently influence the recurrence time of earthquakes. We investigated the porosity distribution in the New
- 23 Zealand's Alpine Fault core in samples recovered during the first phase of the Deep Fault Drilling Project (DFDP-
- 24 1B) by using two-dimensional nanoscale and three-dimensional microscale imaging. Synchrotron X-ray
- 25 microtomography-derived analyses of open pore spaces show total microscale porosities in the range of 0.1 to
- 26 0.24%. These pores have mainly non-spherical, elongated, flat shapes and show subtle bipolar orientation.
- 27 Transmission electron microscopy reveals that nanoscale pores ornament grain boundaries of the gouge material,
- especially clay minerals. Our data implies that: (i) the distribution of clay minerals controls the shape and orientation
- 29 of the associated pores; (ii) porosity was reduced due to pressure solution processes; and (iii) mineral precipitation in
- 30 fluid-filled pores can affect the mechanical behaviour of the Alpine Fault by decreasing the already critically low
- 31 total porosity of the fault core, causing elevated pore fluid pressuresfluid overpressure, and/or introducing weak
- 32 mineral phases, and thus lowering the overall fault frictional strength. We conclude that the current state of porosity
- in the Alpine Fault core is likely to play a key role in the initiation of the next fault rupture.

## 1. Introduction

from fault cores can play a key role in fault slip.

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- Fault mechanics, fault structure and fluid flow properties of damaged fault rocks are intimately related (Gratier and Gueydan, 2007; Faulkner et al., 2010). Fault rupture is associated with intense brittle fracturing that enhances porosity, and thus permeability, and therefore also possible rates and directions of fluid propagation within fault zones (Girault et al., 2018). Conversely, post seismic recovery mechanisms (gouge compaction and pressure solution processes) result in porosity, permeability and fluid flow propagation reductions (Renard et al., 2000; Faulkner et al., 2010; Sutherland et al., 2012). These processes may cause elevated pore fluid pressures within fault cores, and trigger frictional failure (Sibson, 1990; Gratier et al., 2003). Therefore, the state of porosity within rocks
- 43 The Alpine Fault of New Zealand is late in its seismic cycle (Cochran et al., 2017), so studying it allows us to 44 investigate pre-earthquake conditions that may influence earthquake nucleation and rupture processes. Recently, 45 drilling operations were undertaken in this fault zone to investigate the in situ conditions (Sutherland et al, 2012, 46 2017). Slug tests in the DFDP-1B borehole (Sutherland et al., 2012) and laboratory permeability measurements of 47 core samples (Carpenter et al., 2014) indicate permeability decreases by six orders of magnitude with increasing 48 proximity to the fault. Furthermore, Sutherland et al. (2012) documented a 0.53 MPa fluid pressure difference across 49 the principal slip zone (PSZ) of the fault, which suggests that the fault core has significantly lower permeability than 50 the surrounding cataclasite units. It is therefore interpreted to act as a fault seal that limits fluid circulation within its 51 hanging wall (Sutherland et al., 2012). Permeability variations like this are closely associated with the porosity 52 evolution of fault cores, and thus are likely to affect the fault strength and seismic properties (Sibson, 1990; Renard 53 et al., 2000; Gratier and Gueydan, 2007).
- In this study, we investigate the porosity distribution in rocks from the Alpine Fault core and consider the potential effects of this porosity on fault strength. We have measured open pore spaces in these rocks from X-ray computed tomography (XCT) datasets and examined pore morphology by implementing quantitative shape analyses and using transmission electron microscopy (TEM).

## 2. Geological setting

New Zealand's Alpine Fault (Fig. 1a) is a major active crustal-scale structure that ruptures in a large earthquake every 291 ± 23 years, the last one of which occurred in 1717 (Cochran et al., 2017). The fault is the main constituent of the oblique transform boundary between the Australian Plate and the Pacific Plate, accommodating around 75% of the relative plate motion. Ongoing dextral strike-slip at 27 ± 5 mm yr<sup>-1</sup> along the fault has resulted in a total strike-separation of ~ 480 km over the last 25 Ma (Norris and Cooper, 1995, 2001; Norris and Toy, 2014). In Neogene time, a dip-slip component added to the fault motion has resulted in more than 20 km of vertical uplift of the hanging wall (Norris and Cooper, 1995, 2001; Norris and Toy, 2014). Consequently, rocks comprising the hanging wall of the fault have been exposed in various outcrops, where they can be studied in detail. The amphibolite facies Alpine Schist is the metamorphic protolith of a ~ 1 km thick mylonite zone, which has been exhumed from depth and now structurally overlies an up to 50 m thick zone of brittlely deformed cataclasites and

gouges (e.g. Norris and Cooper, 1995, 2001; Norris and Toy, 2014). These rocks have been investigated in outcrops and from samples collected in three boreholes during the two phases of the Deep Fault Drilling Project (DFDP-1A,

DFDP-1B and DFDP-2B; Fig. 1a) along the Alpine Fault (Sutherland et al., 2012; Toy et al., 2015; Toy et al., 2017).

Most of the brittle shear displacement along the fault has been accommodated within the fault core, which includes Principal Slip Zone (PSZ) gouges and cataclasite-series rocks (Toy et al., 2015). Both in surface outcrops and drill core samples, the Alpine Fault manifests as a thin (5 to 20 cm thick) gouge zone with a predominantly random fabric of clay-rich material (Toy et al., 2015; Schuck et al., 2020). This cohesive but uncemented layer has a significantly finer grain size than the surrounding cataclasite units, which shows that the material was reworked only within this layer, most probably as a result of ultracomminution due to multiple shear events under brittle conditions (Boulton et al., 2012; Toy et al., 2015). The local presence of authigenic smectite clays (Schleicher et al., 2015) and calcite and/or chlorite mineralization within sealed fractures and in the gouge matrix (Williams et al, 2017) indicate that mineral reactions are restricted to an alteration zone within the fault core (Sutherland et al., 2012; Schuck et al., 2020). The Alpine Fault core has been interpreted to have formed during a cyclical history of mineralization, shear, and fragmentation (Toy et al., 2015). In addition, in the DFDP-1B borehole (Fig. 1b, Sutherland et al., 2012) fault gouges occur at two distinct depths: 128.1 m (PSZ-1) and 143.85 m (PSZ-2), which shows that the slip was not localized within a single gouge layer (Toy et al., 2015).

## 3. Sample description and analytical methods

# 3.1 Samples

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- 87 Porosity analyses were performed on four samples representing PSZ gouges and cataclasites of the Alpine Fault
- core, which were recovered from the DFDP-1B borehole (Fig. 1b, c; Sutherland et al., 2012). These are DFDP-1B
- 89 58\_1.9, DFDP-1B 69\_2.48, DFDP-1B 69\_2.54 and DFDP-1B 69\_2.57. Sample nomenclature includes drill core run
- 90 number, section number, and centimeters measured from the top of each section. These samples were recovered
- 91 from drilled depth of 126.94 m, 143.82 m, 143.88 m and 143.91 m, respectively.
- 92 Detailed lithological and microstructural descriptions of the DFDP-1B drill core were carried out simultaneously
- 93 with, and after the drilling operations by the DFDP-1 Science Team, and these data were later summarized by Toy et
- 94 al. (2015). Samples DFDP-1B 58\_1.9 and DFDP-1B 69\_2.48 belong to the upper foliated cataclasite units (Fig. 1b,
- 95 c; Toy et al., 2015). These were described as ultracataclasites with gouge-filled shears located above PSZ-1 and
- 96 PSZ-2 respectively. Sample DFDP-1B 69\_2.54 represents the gouge layer that defines PSZ-2, whereas sample
- 97 DFDP-1B 69\_2.57 is composed of brown ultracataclasites that belong to the lower cataclasite unit (Fig. 1b, c; Toy et
- 98 al., 2015).

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## 3.2 X-ray computed tomography (XCT)

- We imaged the samples using absorption tomography, where the signal intensity depends on how electron density
- and bulk density attenuate a monochromatic X-ray along its path through the material (e.g. Fusseis et al. 2014). We
- acquired the X-ray microtomography data for this study at the 2-BM beamline of the Advanced Photon Source,

Argonne National Laboratories USA in December 2012. The non-cylindrical samples of ~7 mm height and ~4 mm diameter were drilled parallel to the foliation and mounted on a rotary stage and imaged with a beam energy of 20 keV. A charge-couple device camera collected images at 0.25° rotation steps over 180° at- sample detector distance of 70 mm and FoV 2.81 mm. The voxel size was 1.3 μm. We have reconstructed the datasets with a filtered back-projection parallel beam reconstruction into 32-bit gray level volumes consisting of 2083 \* 2083 \* 2083 voxels using X-TRACT (Gureyev et al., 2011).

## 3.3 Analyses of XCT datasets

Data analyses and image processing were performed using the commercial software package Avizo 9.1 (Fig. 2). Initially, the datasets were rescaled to 8-bit grey scale volumes for enhanced computer performance. In addition, small volumes of interest were cropped from the whole volume before a non-local means filter was applied to reduce noise (Buades et al., 2005). On the filtered gray-scale images, porosity was identified as the darkest phase (Fig. 2a). The corresponding gray-scale values were thresholded, and the datasets were converted into binary form. However, this threshold range also captured cracks within a sample, which are likely to result from depressurization during core recovery (Fig. 2b). To omit the cracks, thresholded components with volumes larger than the volume of 200 face connected voxels (439.4 µm³) were excluded from the binary label images by using the morphological operation 'connected components' built in software Avizo 9.1. Clusters of connected components were then created to visualize 3D volumes of segmented pore spaces (Fig. 2c).

Unfortunately, this methodology results in either loss of larger pores or inclusion of small cracks depending on the implemented limit of connected components, and thus calculating total porosities includes significant bias. Therefore, "connected components" limited up to 200 connected voxels were used only for visualization purposes. Instead, the volumes and shape characteristics of segmented materials (including cracks) were exported from Avizo software in numerical format, and volume distributions within a sample were plotted on a logarithmic scale in Matlab (Fig. 3). Data up to a specific volume size were fit to a polynomial curve, and then the curve was extrapolated to the X intercept, which is the expected maximum pore size (Fig. 3). Total porosities were then estimated by integrating the curve, which excludes all volumes on the right side of the curve. Total porosities are presented as a percentage of the whole sample volume (Fig. 3). The implemented equations are presented in Supplementary material 1.

Pore shapes were analyzed on bivariate histograms plotted on Matlab by using the numerical pore characteristics, previously extracted from Avizo software. Only pore volumes between 21.97 µm³ (10 voxels) and 878.8 µm³ (400 voxels) were included to avoid bias in the data due to insufficient voxel count and presence of cracks, respectively. Individual pores in our dataset are separated (Fig. 2c), thus For each pore, the covariance matrix of the volume each pore was calculated, and the three eigenvalues of this covariance matrix were extracted. These three values correspond to the three main orthogonal directions in each pore (i.e. the longest, medium and shortest axes) and we use them as proxies to describe pore geometry. Thus, their amplitudes provide information on the spatial extension of the pore and its shape. The ratio between the medium and largest eigenvalues of each pore defines its elongation

- 138 (Fig. 4), the ratio between the smallest to the largest its sphericity (Fig. 5), and the ratio of the smallest to the
- medium its flatness (Fig. 6).
- The angles  $\theta$  and  $\phi$  that describe the orientation of the longest axis of each pore with respect to the main axis of the
- 141 3D scan were calculated. These angles were translated into trend and plunge and then plotted on a lower hemisphere
- 142 equal area stereographic projection with a probability density contour to display the distribution of pore unit
- orientations (Fig. 7).

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## 3.4 Transmission electron microscopy (TEM)

- High resolution—TEM images were collected on a FEI Tecnai G2 F20 X-Twin transmission electron microscope,
- located at the German Research Centre for Geosciences (GFZ), Potsdam, Germany (Fig. 8). The instrument is
- equipped with field-emission gun (FEG) electron source and high-angle annular dark-field (HAADF) Detector.
- 148 Images were collected from samples placed on a Gatan double-tilt holder at 200kV. TEM sample preparation was
- performed with focused ion beam (FIB) milling at GFZ Potsdam using a HELIOS system operated at 30 kV.

#### 150 **4. Results**

## 4.1 XCT-derived characteristics of porosity

- All samples contain low total porosities, ranging from 0.1% to 0.24% (Fig. 3). However, it should be noted that the
- lower cataclasite sample (DFDP-1B 69 2.57) has twice as much pore space (Fig. 3d) as any of the other samples.
- The characterized pore size distributions range over almost three orders of magnitude for all samples (Fig. 3).
- Furthermore, the expected maximum pore size volume was estimated to be largest in the PSZ-2 sample (DFDP-1B
- 156 69 2.54), reaching 862 μm<sup>3</sup> (Fig. 3c).
- 157 In all samples, shape analyses of pores with volumes between 21.97 μm<sup>3</sup> (10 voxels) and 878.8 μm<sup>3</sup> (400 voxels)
- demonstrate predominantly elongated (Fig. 4), non-spherical (Fig. 5) and flat pore shapes (Fig. 6). This is
- particularly pronounced for the smaller pore volumes. The number of elongated pores per sample is increasing in the
- upper foliated cataclasites (Fig. 4a and b) with increasing proximity to PSZ-2, where most elongated pores occur
- 161 (Fig. 4c). Conversely, the lower cataclasite sample demonstrates proportionally fewer elongated pores within the
- sample (Fig. 4d). The degree of sphericity is uniform for all samples, and pores appear as mainly non-spherical (Fig.
- 163 5). Few isolated spherical pores are manifested only by small pore volumes (Fig. 5). A trend of increasing the
- number of flat pores is observed with increasing sample depth (Fig. 6), and most flat pores are detected in the lower
- cataclasite (Fig. 6d).
- The orientations of the individual pore units show two distinctive peaks with opposite vergence, defining bipolar
- distributions of pore orientations (Fig. 7). The observed bipolarity is subtle in samples DFDP-1B 58\_1.9 (Fig. 7a)
- and DFDP-1B 69\_2.48 (Fig. 7b), and more obvious in samples DFDP-1B 69\_2.54 (Fig. 7c) and DFDP-1B 69\_2.57
- 169 (Fig. 7d).

## 4.2 Microstructural characteristics of porosity

- 171 TEM characterization of the gouge material from PSZ-2 (sample DFDP-1B 69\_2.54) reveals that the Alpine Fault
- gouges have composition, are composed of comprising angular quartz and/or feldspar fragments (~200 nm in size),
- wrapped by smaller phyllosilicates (< 100 nm long). This random fabric is ornamented by nanoscale pores (< 50
- nm), distributed along all grain and phase boundaries, especially abundant along clay minerals (Fig. 8a).
- The gouge material also demonstrates phyllosilicate-rich areas, defined by an increase in the clay/clast ratio. In these
- zones, fine (< 100 nm long) and coarser (few µm long) clay grains coexist and are aligned in wavy fabric that
- surrounds sporadic protolith fragments (Fig. 8b). Pore spaces are again distributed along the boundaries of the
- 178 constituent mineral grains but some of them are larger (~0.5 µm) withand thin ellipsoidal or elongated shapes (Fig.
- 8b, c). These pores are commonly associated with inter-clay layer porosity. Large size pores are also observed along
- 180 quartz-feldspar grains, where pores are associated with multiple grains and occasionally disrupt grain boundaries,
- thus were labelled asas cracks along boundaries of quartz and/or feldspar grains (i.e. fracture porosity; (Fig. 8d).

## **5. Discussion**

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## 5.1 Characteristics of porosity within the Alpine Fault core

- Porosity analyses of samples from, or in close proximity to the two PSZs encountered in the DFDP-1B drill core
- reveal total pore volumes between ~ 0.1 and 0.24% (Fig. 3). These values are significantly lower than the porosity
- estimates from other active faults in the world, such as: 0.2 to 5.7% total porosity in the core of the Nojima Fault,
- Japan (Surma et al., 2003) and 0 to 18% in the San Andreas Fault core (Blackburn et al., 2009). The Alpine Fault
- core contains total pore space volumes, comparable only with the lower porosities in these previous studies. It
- should be noted that the smallest pore spaces captured in the XCT datasets are 1.3 µm in size due to resolution
- 190 <u>acquisition</u> constrains, whereas nanoscale porosity was identified on the TEM images. Therefore, the estimated total
- porosities represent only minimum values of the open pore spaces in the Alpine Fault core. However, the addition of
- 192 nanoscale porosity volumes is unlikely to dramatically affect the final total porosity of these rocks because they
- 193 comprise a very small total volume.
- TEM images presented here mainly focus on nano-scale materials (Fig. 8a, c, d) but were also used to describe the
- distribution of micro-porosity in these rocks (Figure 8b). The pores visible on grain and phase boundaries in On
- figure 8b-pores have similar sizes comparable to the small range of pores segmented on XCT images (> 1.3 μm in
- diameter), and thus we conclude that this is the typical habit of both nano- and micro-pores within the Alpine Fault
- 198 core are distributed on grain and phase boundaries, especially of clay minerals (Fig. 8). In addition, both quantitative
- micro-porosity shape analyses (Fig. 4, 5 and 6) and nano-pores identified on TEM images (Fig. 8) reveal that a
- significant population of pores are predominantly non-spherical with elongated, flat shapes. We attribute this
- 201 observation to the tendency of these pores to ornament clay minerals where pores are attained and elongated along
- 202 their (001) planes (Fig. 8b, c and d).

Foliation in the upper cataclasites is defined by clay-sized phyllosilicates, that become more abundant with proximity to the PSZ (Toy et al., 2015), where weak clay fabric is developed (Schleicher et al., 2015). This gradual enrichment in clay minerals coincides with the subtle development of bipolar distributions of pore orientations with increasing sample depth (Fig. 7). This observation and the fact that pores are mainly attained along grain boundaries of clays (Fig. 8) suggest that the distribution of clay minerals also controls pore orientations within the Alpine Fault core. Previously, the phyllosilicate foliation in the Alpine Fault cataclasites has been used to define shear direction (Toy et al., 2015). Thus, we speculate that pore orientations in these rocks are also systematically related to the kinematic framework of the shear zone. If these pores represent remnants of fluid channels, their spatial orientation is likely to reflect the fluid flow directions during deformation. To address this possibility more data for systematic analyses of pore orientations are needed.

# 5.2 Porosity reduction within the Alpine Fault core

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- The comparatively lower porosity estimates of the Alpine Fault core than other active faults (e.g. the Nojima Fault, Surma et al., 2003, and the San Andreas Fault, Blackburn et al., 2009) can be attributed to the fact that the Alpine Fault is late in its seismic cycle and the last seismic event occurred in 1717 (Cochran et al., 2017). Porosity of the fault core is considered to evolve during the seismic cycle when fault rupture can cause porosity increase up to 10% (Marone et al., 1990), and the consequent healing mechanisms lead to porosity decrease over time due to mechanical compaction of the fault gouge and/or elimination of pore spaces within the fault core due to pressure solution 220 processes (Sibson, 1990; Renard et al., 2000; Faulkner et al., 2010).\_-TEM data presented here show abundance of newly precipitated authigenic clays, wrapped around coarser clay minerals (Fig. 8b). Furthermore, delicate clay 222 minerals form fringe structures (Fig. 8a), and strain shadows (Fig. 8c) around larger quartz-feldspar grains. These microstructural observations demonstrate that pressure solution processes operated within these rocks (Toy et al., 2015).
  - Evidence for pressure solution processes has been previously documented in all units, comprising the Alpine Fault core (Toy et al., 2015). Abundant precipitation of alteration minerals (Sutherland et al., 2012), calcite filled intragranular and cross-cutting veins (Williams et al., 2017), and the occurrence of newly formed smectite clays (Schleicher et al., 2015) indicate extensive fluid-rock reactions. In addition, anastomosing networks of opaque minerals (such as graphite; Kirilova et al., 2017), which define foliation in the upper cataclasites (Toy et al., 2015), have been interpreted to be concentrated by pressure solution processes during aseismic creep (Toy et al., 2015; Gratier et al., 2011). The petrological characteristics of the Alpine Fault core lithologies identify solution transfer mechanisms likely were the dominant mechanism for pore closure within these rocks.
  - Post-rupture porosity reduction is known to operate three to four times faster within fine-grained fault gouges than in coarser-grained cataclasites (Walder and Nur, 1984; Sleep and Blanpied, 1992; Renard et al., 2000), which may explain the differences in total porosity between the gouge-containing samples and the footwall ultracataclasite – DFDP-1B 69-2.57 (Fig. 3). Furthermore, previous studies documented less carbonate and phyllosilicate filling of cracks in the Alpine Fault footwall cataclasites as compared to the hanging wall cataclasites (Sutherland et al., 2012;

- Toy et al., 2015), suggesting more reactive fluids are present and isolated withing the hanging wall of the Alpine
- Fault. Thus, more intense dissolution-precipitation processes took place in the fault's hanging wall, which very
- likely resulted in more efficient porosity reduction, as demonstrated by our porosity estimates (Fig. 3).
- 241 As aforementioned, porosity reduction is known to increase with time after an earthquake event due to post rupture
- 242 healing mechanisms (Sibson, 1990; Renard et al., 2000; Faulkner et al., 2010). Thus, the comparatively lower
- 243 porosity estimates of the Alpine Fault core than other active faults (e.g. the Nojima Fault, Surma et al., 2003, and the
- 244 San Andreas Fault, Blackburn et al., 2009) can be attributed to the fact that the Alpine Fault is late in its seismic
- 245 eyele (Cochran et al., 2017).

## 5.3 Effects of porosity on the Alpine Fault strength

- The extremely low porosity estimates presented here (Fig. 3) are consistent with the low permeabilities of 10<sup>-18</sup> m<sup>2</sup>
- measured experimentally in clay-rich cataclasites and gouges from the Alpine Fault zone (Carpenter et al., 2014). In
- addition, the documented difference of total porosities between the hanging wall and footwall samples (Fig. 3)
- 250 implies different intensity of pressure solution processes, and thus compartmented fluid propagation. Our data thus
- 251 provide independent verification is comparable with of the permeability measurements in that study (Carpenter et
- al., 2014) and <u>yields</u> increased confidence in their interpretation of a permeability gradient with distance from the
- PSZ, which itself acts as a hydraulic seal (Sutherland, et al., 2012). The existence of such a barrier to flow is
- characteristic for faults undergoing creep and locked faults (Rice, 1992; Labaume et al., 1997; Wiersberg and
- Erzinger, 2008). However, much higher permeabilities in the surrounding damaged rocks (Carpenter et al., 2014)
- allow fast propagation of fluids within them and can cause localization of high fluid pressures on one side or the
- other of a hydraulic seal (Sibson, 1990). Such fluid pressures can enhance gouge compaction and pressure solution
- 258 processes within the fault core, which will eventually introduce zones of weakness and thus may trigger fault slip
- 259 (Faulkner et al., 2010).
- 260 Previous studies and the observations presented here show that fluids were present in the Alpine Fault rocks. Fluid-
- filled pores represent a favorable environment for mineral precipitation, which can affect the fault strength in two
- ways: (i) V-very small decrease of these critically low total porosities due to mineral precipitation would cause fluid
- pressurization, which is a well-known fault weakening mechanism described by (Byerlee, 1990; and Sibson, 1990).
- However, this pressure increase will be slightly offset by inclusion of fluids into new hydrous minerals; (ii)
- deposition of frictionally weak phases (such as clay minerals and graphite), especially if they decorate grain contacts
- and/or form interlinked weak layers, would lower the overall frictional strength (Rutter et al., 1976; Niemeijer et al.,
- 267 2010).
- 268 Precipitation of authigenic clay minerals was identified on our TEM data (Fig. 8) and also documented by previous
- studies (Schleicher et al., 2015). As well as having low frictional strengths (Moore and Lockner, 2004), clay
- 270 minerals may also contribute to the formation of an impermeable seal if they form an aligned fabric, and thus can
- enhance the likelihood of fluid-pressurization in the fault rocks (Rice, 1992; Faulkner et al., 2010). In addition,
- graphite, which was previously documented in these rocks by previous studies (Kirilova et. al., 2017), may

- effectively weaken the fault due to mechanical smearing (Rutter et al., 2013) and/or localized precipitation within
- 274 strained areas (Upton and Craw, 2008). Such graphite precipitation within shear surfaces was previously
- documented by Kirilova et al. (2017).
- In summary, the presence of trapped fluids in the low porosity rocks of the Alpine Fault core possibly controls the
- 277 mechanical behavior of the fault and could be responsible for future rupture initiation due to fluid pressurization
- and/or precipitation of weak mineral phases. This hypothesis is further supported by an experimental study showing
- 279 that the DFDP-1 gouges are frictionally strong in the absence of elevated fluid pressure (Boulton et al., 2014).

#### 6. Conclusions

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- Analyses of XCT-datasets and TEM images of borehole samples from the core of the Alpine Fault reveal micro- and
- nanoscale pores, distributed along grain boundaries of the constituent mineral phases, especially clay minerals. The
- 283 tendency of these pores to ornament clays defines their predominantly non-spherical, elongated, flat shapes and the
- bipolar distribution of pore orientations. The documented extremely low total porosities (from 0.1 to 0.24 %) in
- these rocks suggest effective porosity reduction. Microstructural observations presented here and documented in
- previous studies indicate that pressure solution processes were the dominant healing mechanism, and that fluids
- were present in these rocks. Therefore, fluid-filled pores may be places where fluid overpressures elevated pore fluid
- 288 <u>pressures</u> develop, due to further mineral precipitation that decreases the already critically low total porosities.
- Alternatively, they may also facilitate deposition of weak mineral phases (such as clay minerals and graphite) that
- may very effectively weaken the fault. We conclude that the current state of the fault core porosity is possibly a
- controlling factor on the mechanical behaviour of the Alpine Fault and will likely play a key role in the initiation of
- the next fault rupture.

# Data availability.

Matlab code and numerical data of pore volumes can be found in Supplementary material 1.

## **Authors contribution**

- 296 Kirilova reconstructed, processed, and analysed the XCT datasets presented here, interpreted the TEM data and
- 297 prepared the manuscript. Most of this work was performed during Kirilova's PhD under the academic guidance of
- 298 Toy. Toy and Gessner collected the XCT data with technical support by Xiao. Renard and Sauer contributed with
- valuable discussion about XCT data analyses. Wirth enabled TEM data acquisition and provided his expertise on
- TEM data interpretation. The final version of this manuscript benefits from collective intellectual input.

## **Competing interests**

The authors declare that they have no conflict of interest.

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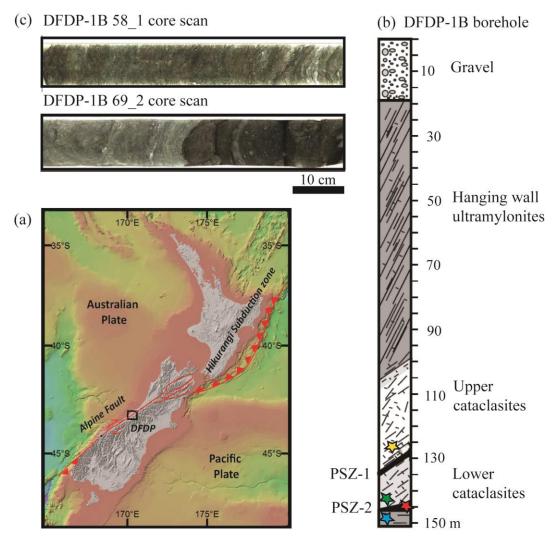
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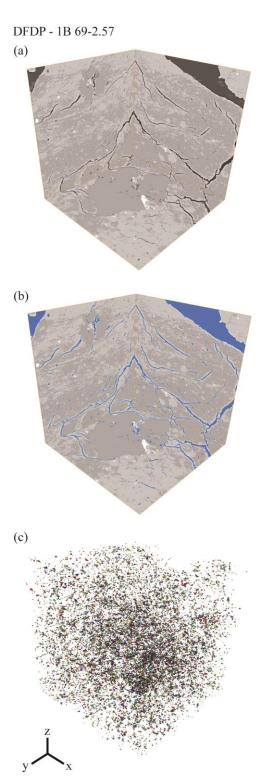
# 421 Figures

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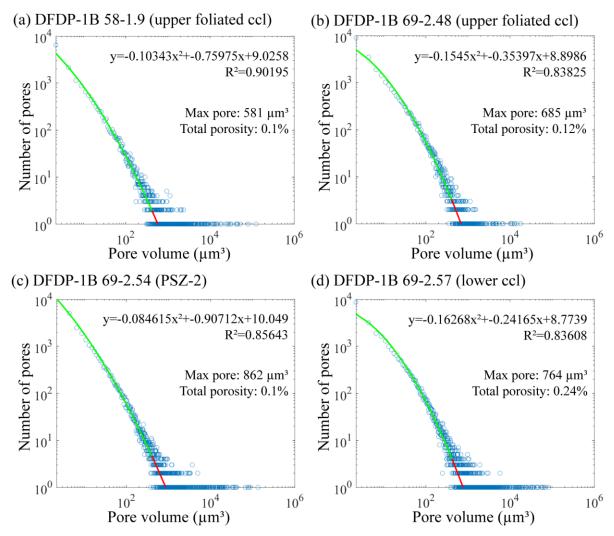
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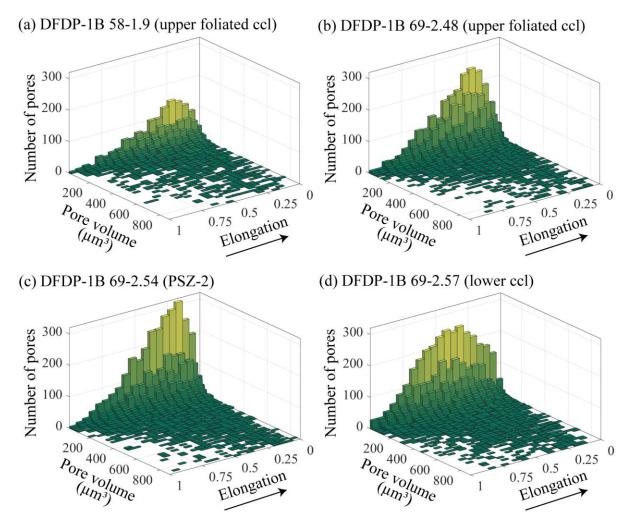
**Figure 1.** (a) Location map of DFDP drill sites (a bathymetric map compiled by NIWA). <u>Drill site coordinates:</u> 43°17′5″S, 170°24′22″E (b) Schematic diagram of the sampled lithologies in DFDP-1B borehole (modified after Sutherland et al., 2012). (c) Scans of DFDP-1B drill core. Samples were collected from the locations indicated with stars: yellow – DFDP-1B 58\_1.9; green – DFDP-1B 69\_2.48; red – DFDP-1B 69\_2.54; blue – DFDP-1B 69\_2.57.



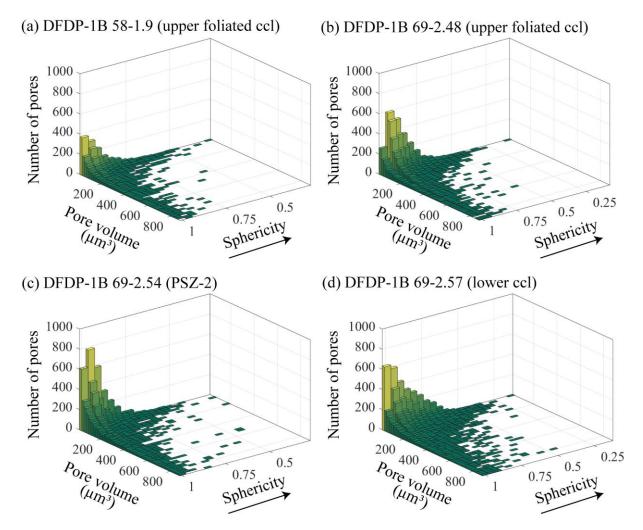
**Figure 2.** X-ray tomography data processing workflow. (a) Gray scale images in xy, xz and yz directions (b) Threshold of the darkest gray scale phase in each sample, corresponding to voids (pores and fractures); (c) 3D volume of the segmented pore spaces after <u>removal of</u> the fractures due to sample decompaction and coring damaging effects were removed.



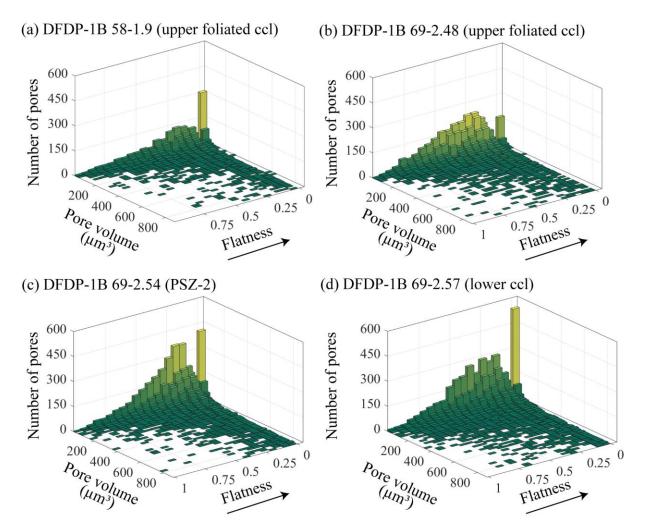
**Figure 3.** Plots of pore volume versus number of pores for each sample. Estimates of total porosity and size of the maximum expected pore are also shown, as well as the curve fitting function for each dataset.



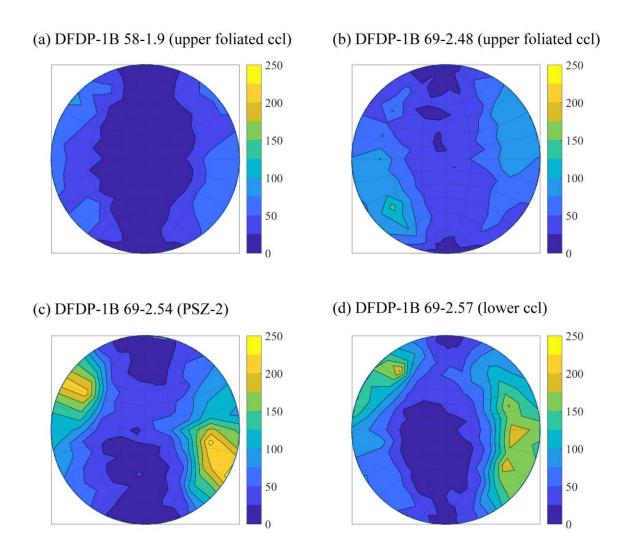
**Figure 4.** Bivariate histograms showing elongation versus pore volume ( $\mu$ m<sup>3</sup>) and number of pores for each sample. The arrow indicates the direction of increasing elongation.



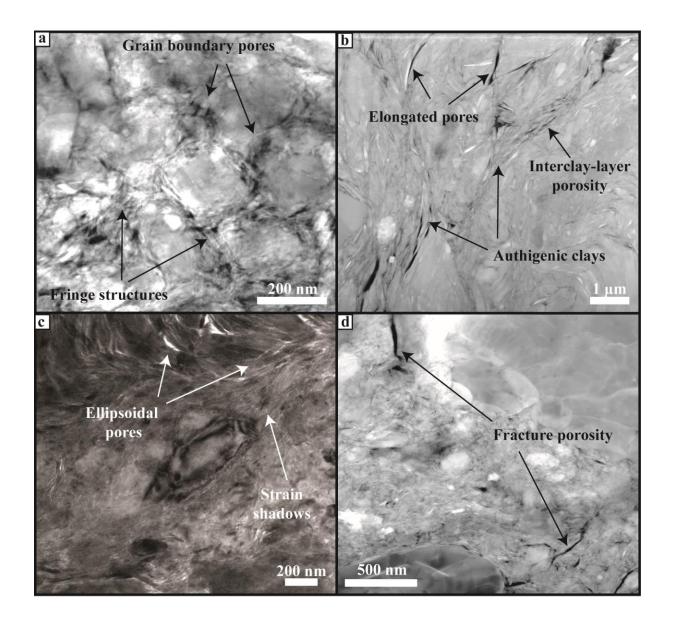
**Figure 5.** Bivariate histograms showing sphericity versus pore volume ( $\mu$ m<sup>3</sup>) and number of pores for each sample. The arrow indicates the direction of increasing sphericity.



**Figure 6.** Bivariate histograms showing flatness versus pore volume ( $\mu m^3$ ) and number of pores for each sample. The arrow indicates the direction of increasing flatness.



**Figure 7.** Distribution of pore unit orientations plotted on a lower hemisphere equal area stereographic projection with a probability density contour.



**Figure 8.** Transmission electron microscopy images collected from the gouge sample DFDP-1B 69\_2.54 (PSZ-2). (a) and (c) are bright-field images, where porosity appears as bright contrast areas. (b) and (d) are high-angle annular dark field images, where pores appear as dark contrasts areas. (a) TEM bright-field image of homogeneous fault gouge area. Quartz/feldspar grains, wrapped by fine authigenic clays, displaying fringe structures. Pores with subangular shape distributed along grain boundaries. (b) HAADF image of phyllosilicate-rich gouge area. Co-existence of fine authigenic clays with coarser clay mineral grains. Elongated pores and interlayer porosity. (c)TEM bright-filed image of ellipsoidal pores in phyllosilicate-rich areas. Examples of strain shadows along quartz/feldspar grains. (d) HAADF image of fracture porosity along grain boundaries of quartz/feldspar grains.