A new X-ray transparent flow-through reaction cell for a μ-CT- based concomitant surveillance of the reaction progress of hydrothermal mineral-fluid interactions

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The manuscript presents a new X-ray transparent cell concept for reactive transport processes of reservoir rocks. The topic of the paper is very interesting as XRCT (X-Ray Computed microTomography) is well available in our days and advanced imaging techniques give a great inside into morphology evolution for complex processes like the one mentioned here. The content of the manuscript fits therefore very well in the Special Issue of SE.

Nevertheless, my impression is that various important aspects keep unclear in the manuscript. Besides various missing technical details, the reviewer mainly misses detailed images and/or technical drawing explaining the cell concept and the underlying „imaging" procedure. Nevertheless, this could be easily improved in a revised version. The reviewer is looking forward to that version!

Recommendation: Major Revision

Remarks in detail:

- To discuss differences of your cell concept compared to others available in the literature for similar applications, it would be very important to cite additional papers, where X-ray transparent cells and/or feasibility studies have been discussed extensively, cf. [1,2] and others. Further, a critical discussion about advantages/disadvantages of your concept would be very much appreciated!
- Page -2-, Line 7:
In your mentioned workflow, all bench-top and synchrotron microtomography facilities are meant to be X-ray-based. This should be clarified. You could write e.g.: bench-top and synchrotron X-ray computed microtomography: micro-CT.
- Please check carefully the reference list (typos), i.e. replace Kesten with Kersten
- Page -2-, Line 7:
Why do you call mercury porosimetry a pure statistical consideration which is inferior to micro-CT? Mercury porosimetry is based on pore-scale physics (Washburn-Equation). On the one hand, it is true, that only an averaged pore size could be determined but on the the other hand, the spatial resolution of mercury porosimetry (<< 1 micrometer) is (much) higher then the resolution of various imaging techniques, like e.g. bench-top microtomography. Please clarify!
- Please provide materials properties of PEEK; this could be helpful for the reader. Further, I would suggest to discuss/compare X-ray transparency of PEEK especially w.r.t. alternative materials used for X-ray transparent cells (quartz glass, pure aluminium, carbon,…)
- It would be interesting for the reader if you share more advanced drawings of your cell and of the underlying concept. Perhaps you could provide CAD drawing (supplementary data or explosion sketch for mounting purposes) or detailed drawing of the sealing part? Further, a detailed list of parts (fittings, o-rings, etc) would also be of great interest. In detail: I miss especially a detailed explanation of the (upper) sealing part of the cell. I understand that the dumbbell part is mounted via o-rings to the upper part of the cell. How are the end-caps connected to the fluid lines? I see two 10-32 fingertight fittings, which I could not understand form the drawing. Do you use a dynamometric key to fix the upper part of the cell to the dumbbell part?

- Page -3-, Line 28:
  You mean perhaps: 210 mm in height and 100 mm in diameter?
- Please provide more information about the used back pressure regulator (type, manufacturer, etc)
- How do you measure temperature when the sample is removed from the oven for imaging purposes? You do not have any thermocouple inside the cell?
- Please provide more details about the pressure sensors (type, pressure range, active/passive sensor). How large is the dead volume of the sensors? Have you thought about novel in-line pressure sensors used for instance in micro-fluidic devices?
- Could you provide more information about the dead volume of the total system (incl. dead volume of pressure sensor, valves, end-caps, etc) especially compared to pore volume? This could be of great importance when you change the fluid…
- Page -4-, Line 16:
  It is very interesting and also quite important, that fluid could be extracted from the sample during the experiment. How exactly could that be done in your case? Could you guarantee (during the fluid sampling procedure) that the pore pressure state is kept constant? You are measuring pore pressure, please try to provide information.
- I understand, that the flow-through experiment is in the oven and only removed for imaging purposes. If this is done various times, how do you solve the registration problem of the cell? You are working with a voxel size of 8.95 micrometer, cf. Fig. 3). Thus, you need a higher precision for registration. Does that causes problems?
- Page -4-, Line 29:
  Please describe in detail what you mean with partially saturated with water. How do you inject water in order to guarantee a partial saturation condition? Before injecting water, is the sample saturated with air? Do you flush the sample with CO2? Do you apply a vacuum? I think, even in a feasibility study this workflow information is important.
- Page -6-
  I understand, that you would like to present a feasibility study discussing especially the cell and the flow-through concept for imaging. But even with this goal, I have a further point which could be included in the manuscript without much effort: During the flow-through experiment you measure the pressure gradient (please provide information…) according to Fig. 2. In addition, the flow-through experiment is fluid-flux-controlled. Thus, you could easily calculate permeability and even more, you know temporal permeability evolution through the time period of the experiment. Wouldn't it be interesting, especially in a feasibility study, to compare the exp. obtained permeability evolution with calculated permeability evolution from porosity evolution (i.e. Kozeny-Carman - which you got through the micro-CT snapshots)?

References:

1. Fusseis, F., Steeb, H., Xiao, X., Zhu, W.-L., Butler, I. B., Elphick, S., & Mäder, U. (2014). A low-cost X-ray-transparent experimental cell for synchrotron-based X-ray microtomography studies under geological reservoir conditions. J Synchrotron Rad, 21, 251–253. http://doi.org/10.1107/S1600577513026969/pp5039sup2.zip
2. Ott, H., de Kloe, K., van Bakel, M., Vos, F., van Pelt, A., Legerstee, P., et al. (2012). Core-flood experiment for transport of reactive fluids in rocks. Review of Scientific Instruments, 83(8), 084501. http://doi.org/10.1063/1.4746997