

## ***Interactive comment on “Pore formation during dehydration of polycrystalline gypsum observed and quantified in a time-series synchrotron radiation based X-ray micro-tomography experiment” by F. Fusseis et al.***

**H. Milsch (Referee)**

milsch@gfz-potsdam.de

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Summary:

The paper presents results of one gypsum dehydration experiment performed at ambient pressure and 388 K. Concurrently, a series of X-ray micro-tomographic scans was performed to monitor the reaction progress and the evolution of microstructure. To my knowledge this is the first experiment of this kind ever performed providing deepened insight into the time dependent 3D-microstructure of a devolatilizing rock. The raw data

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has been thoroughly processed and further evaluated. Conclusions are drawn on both the mechanism of reaction progress and a number of textural features of the evolving pore space including a quantification of sample porosity. The paper, overall, is well written, organized, and documented. Not least due to the pioneering character of the research undertaken the paper is certainly suitable for publication in Solid Earth (SE). However, some minor remarks are appended below that the authors should consider to improve their paper.

General comments:

From my own experiences the material used is fairly homogeneous in a way that the reproducibility of an experiment performed under specific conditions is excellent. The one experiment performed should therefore be representative for the particular set of environmental conditions (e.g. pore and confining pressures, temperature, water vapor partial pressure, drainage etc.) - a fact which should be explicitly stated in the text. In this study the assumption that bassanite is the final solid reaction product is not justified by any measurement. The authors should have a look at Milsch et al (2011) where comparable dehydration experiments were performed under environmentally very similar conditions but on significantly larger samples. We show that in air and even at 388 K gypsum ultimately dehydrates to anhydrite (also cf. McAdie, 1964; Fig. 2). If your sample is still intact and available you may want to weigh it and perform the calculation of reaction progress as outlined in Milsch et al. (2011). Additionally, you can then rather precisely determine the overall sample porosity. You may want report the results in your manuscript. Finally, we have microstructural evidence (unpublished data) that at 398 K there is gypsum decomposition also in what you term the “inner domain”. Besides being wet, Ko et al.’s (1997) samples were dehydrated at this temperature which might explain some of the differences to the interpretations of your experiment that you claim in Section 4.1.

Specific comments:

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859, L. 27: the last sentence should be deleted.

860, L. 18 to 861, L. 18: see comment above.

861, L. 20: check the number! The total volume change is 7% for dehydration to bassanite.

861, L. 26: is this the correct Llana-Fúnez paper you are referring to?

861, L. 29: this depends on the (water) pore pressure relative to the pT-equilibrium curve.

862, L. 17-24: this should be moved to Section 4.

865, L. 9: where is the “length scale” in the equation (L. 10)?

865, L. 23-25: this should be moved to Section 4.

866, L. 28: the difference in density is rather significant.

867, L. 21-23: this should be moved to Section 4.

868, L. 15: you only used one sample.

869, L. 9-27: this is very hard to understand and also should be moved to Section 2 as it is a purely technical description.

870, Eq. 1: there is something wrong with this formula. Check the nomenclature and the units; what is  $r$  in L. 9; how does  $D$  obtained compare to potentially available literature data?

870, L. 26-29: dry gypsum is brittle at ambient pressure and moderate temperatures (Milsch and Scholz, 2005)!

871, L. 3: explain what you mean by “the outer domain”.

871, L. 10: this is a natural rock!

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872, Fig. 9: how important is this figure? Replace it by Fig. 5 in the supplement? Also, L. 17 “Fig. 4” refers to Fig. 5?

872, L. 18-23: the paragraph should be moved to Section 2 as it is a purely technical description.

872, L. 25: what is the “raster effect”?

873, L. 4-28: consider moving technical parts of these two paragraphs to Section 2.

873, L. 20: what do you mean by “migrated through a volume”.

874, L. 19-25: again, you should emphasize that this study deals with unconfined and dry conditions.

875, L. 11: at unconfined conditions gypsum will evidently fail at these internal pore pressures, relaxing them immediately! Also see comment above (861, L. 29).

875, L. 21: what do you mean by “run-away”.

876, L. 4-5: you did not run any fluid expulsion experiment, so what is the point? If you had, you very likely would have observed a smooth expulsion curve at decelerating rate (cf. your Fig. 5b).

876, L. 18-27: Ko et al.’s (1997) experiments were performed under particular drainage conditions and under significant effective pressure on wet gypsum! That very likely is the reason. Wang and Wong (2003) refer to these conditions.

878, L. 20: what about a heterogeneity in the temperature distribution?

880, L. 10: again, what do you mean by “run-away”.

880, L. 13: again, this is very speculative.

Fig. 4: from the caption this figure is hard to understand.

Figs. 5, 6, 7, 8, 10, 11, 12, 14: try at least in some words to explain what one sees!

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Fig. 9: see comment above.

Technical corrections:

Throughout the manuscript the tense use should be reconsidered. This particularly applies to sections that relate to the past (e.g. measurements performed).

Throughout the manuscript, also in figure captions,  $\mu\text{m}$  should be used instead of micron and K instead of degree and min instead of ' or minute.

863, L. 5: Olgaard

863, L. 9: analyze

872, L. 23: "ii" (subscript)

873, L. 7: missing "voxel" behind 151-300.

873, L. 8: "e1" (subscript)

878, L. 17: parts

Fig. 6a and 13: porosity is shown fractional compared to Figs. 4 and 12.

Figs. 7, 8: change "micron<sup>3</sup>"

Fig. 10: is "310-140 voxel" correct? Why green and orange circles for the same voxel interval?

Caption Suppl. Fig. 2: of the experiment

Caption Suppl. Fig. 4: proportions

References:

Ko, S.-c., Olgaard, D. L.; Wong, T.-f. (1997): Generation and maintenance of pore pressure excess in a dehydrating system. 1. Experimental and microstructural observations, *J. Geophys. Res.*, 102 (B1), 825-840.

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McAdie, H. G. (1964): The effect of water vapor upon the dehydration of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ , *Canadian Journal of Chemistry*, 42, 792-801.

Milsch, H.; Scholz, C. H. (2005): Dehydration-induced weakening and fault slip in gypsum: Implications for the faulting process at intermediate depth in subduction zones, *J. Geophys. Res.*, 110, B04202, doi:10.1029/2004JB003324.

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Wang, W. H.; Wong, T.-f. (2003): Effects of reaction kinetics and fluid drainage on the development of pore pressure excess in a dehydrating system, *Tectonophysics*, 370, 227-239, doi:10.1016/S0040-1951(03)00188-4.

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