

## ***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. Fuisseis et al.***

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We thank Dr Trippetta for his fast and thorough review of our manuscript.

Dr Trippetta's criticism hinges on three points: 1) That the link between the text and the figures is weak and there are too many figures. 2) That there are inconsistencies in the use of units. 3) That we present the results from a single experiment only.

ad 1) The paper presents a new approach to investigate gypsum dehydration and the related pore formation experimentally. We determine parameters (such as the exact

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number of pores, their size, volume, shape, etc.) that could never be quantified before. Each of the figures focusses on one parameter, and each figure is discussed in the text. We therefore do not see a lot of room for reducing the total number of figures and we certainly do not consider any of the figures 'useless', as indicated by Dr Trippetta. Indeed, the geometrical characteristics of pore space (pore size distribution, aspect ratios, etc.) reported here are essential, yet so far poorly constrained ingredients for effective medium models commonly employed in rock mechanics. These models calculate the mechanical properties of materials as a function of crack porosity.

Dr Trippetta finds that some of the supplementary figures are more instructive than those presented in the text. Specifically, he mentions Suppl. Figures 1 and 4. We would like to point out that Suppl. Figure 1 shows a representative thin section of Volterra gypsum. The experiment is destructive; it is therefore impossible to show a thin section of the actual sample that was later dehydrated during the experiment. Suppl. Figure 4 shows the proportion of sample area where the reaction is 'in progress' in comparison to the calculated reaction progress assuming that the reaction advances concentrically and comes to completion as soon as the front has passed. It contains very limited information on how dehydration actually evolves.

ad 2) We use SI units throughout the manuscript. However, we found it more convenient to use minutes for the time axes. The analyzed time steps are always the same. There was one reference to °C, which we changed to Kelvin. Porosity is always given in percent and we have updated the figures where this was left unclear.

ad 3) We repeated an experiment that has been conducted many times before. It is now generally well established how alabaster from Volterra responds to heating. We present a new methodological approach that successfully overcomes the black box limitations of previous experiments. We use this approach to document the reaction and porosity production in 4D and in situ. We furthermore target an open question concerning the width of the reaction initiation front, which is linked to the breakdown behavior of gypsum. Our experiment is documented and analyzed in unprecedented detail.

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Our interpretation is firmly based on our observations and in the context provided by published state-of-the-art models. Our study gives important clues on the processes that control the initiation and the progress of the reaction and clarifies what the reaction front looks like.

We believe that these aspects render our results 'suitable for the community'. Although we acknowledge that reproducibility should be warranted, we do not see any indication that, if we repeated the experiment, our conclusions would be significantly different beyond what is already discussed in the text. We therefore see no critical need for further experiments at this stage. The applicability of our conclusions is determined by the boundary conditions of our experiment. A paper that develops the extrapolation of our results to more general conditions is currently in preparation (Karrech et al., in prep.). The degree to which our conclusions can be applied to the diagenesis of natural evaporites remains to be tested, however, this was not the objective of this study.

We would furthermore like to point out the immense costs and efforts that are necessary to conduct an experiment like the one reported in this paper. There are no off-the-shelf solutions available, neither for in-situ heating in a synchrotron x-ray microtomograph, nor for the computationally extremely expensive data analysis. Future experiments will benefit from the infrastructure and routines we developed for the one reported here.

Detailed reply to Dr Trippetta's comments (which are marked by '>')

>Title: In my opinion the title should specify that all the dataset is related to just one sample. . .just as a suggestion I was thinking to something like: Pore formation and evolution during dehydration of polycrystalline gypsum using synchrotron radiation based X-ray micro-tomography: insights from a sample of Volterra gypsum.

We have modified the title to: Pore formation during dehydration of a polycrystalline gypsum sample observed and quantified in a time-series synchrotron X-ray microtomography experiment

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>Organization of the paper: the paragraph 3.2 is subdivided in 2 subparagraphs without any clear needs. I suggest to rearrange as: 3.1 Dehydration initiation 3.2. Porosity evolution 3.3 Drainage architecture evolution

Although the porosity- and the drainage evolution are linked, two different types of analyses are presented in the sub-sections 3.2.1 and 3.2.2. We therefore prefer to keep the existing subdivision.

>Paragraph 4.3 should be a discussion subparagraph, however the discussion of the results are limited to the last 3 rows. This looks more like a discussion on the methodology that, in my opinion is in the wrong position. Since from the adopted methodology derive all the results this discussion should be moved to the introduction paragraph in order to both explaining to the reader the strength of the adopted method and to avoid criticisms on the methodology.

The quantitative analysis of microtomographic data depends to a significant extent on the choice of the thresholding technique. In this section we discuss and justify the choice of our thresholding technique in the context of existing literature. For this we consider the discussion chapter the correct location.

>The sentences immediately after par 4 (pp 874 #20-25) are inappropriate in that position. This is a sort of summary that maybe can be moved to the conclusion paragraph or deleted at all.

The criticized paragraph forms the introduction to the rather comprehensive 'discussion and interpretation' section of our paper. It serves as a link to the introduction and will make reading the paper easier for those who limit their attention to the 'introduction' and 'discussion/interpretation' sections.

>In the paper Kelvin are used whilst in the introduction Celsius are used (p 860 #13).

Corrected.

>Par 2.3 (p864 #14) What "in situ" means? Did you performed your measurements in

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Volterra?

'In situ' means that the dehydration reaction was observed in real time by a non-destructive technique at the place where it occurs, in the Synchrotron x-ray microtomograph. From a technical perspective this means that the specimen was kept in position inside the furnace over most of the experiment. This helped to maintain a stable dehydration environment and minimize the uncertainties introduced by uncontrolled temperature variations. We believe that this should become clear from the text.

>P865 #28 Here are first presented the "nine time steps" never mentioned before.

The nine scans (or time-steps) are mentioned on p864, line 14 and again on p865, lines 1-2.

>P869 #6-11 In this part you cite "supplement fig 3" three times whilst, for example fig. 5b is never cited along the whole paragraph. I strongly suggest to reorganize the order, the number and the choice of the figures.

This paragraph presents details of how an analysis, whose results are reported in the following paragraph (and where Figure 5b is referenced), was conducted. Supplementary Figure 3 shows technical details of how we conducted the measurements and we consider the appendix the right place for the figure.

>P871#23 "the numerical expansion/shrinking experiment"..what are we talking about? Has this experiment ever mentioned before?

This (analytical) exercise, which serves to determine the largest possible error in our measurements, was described in the methodology section, over 11 lines from p867 line 24.

>P872 #27 these observation look very speculative and they are hardly observable in C513 fig 10. Moreover the main point is, why do you need to perform this kind of analysis?

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In our work we certainly take great care to avoid any mixing of observation and 'speculation', or 'interpretation'. In our opinion there are no 'speculative' observations. The data that are described in these lines are clearly shown in Figure 10 and discussed in section 4.2. Determining the shapes of pores is critical for interpreting their genesis as well as for a later characterization of their effects on rock mechanical properties such as permeability, stiffness, conductivity, etc.

>P876 #9 "Figure 4 shows that 50% of the sample area in horizontal cross-section reacts within the first 17min" Fig 4 really shows this (maybe is suppl fig. 4)? Once again 17 minutes has never been used as a time step before.

We have modified this section slightly to make it more comprehensible.

>Figures: Most of the figures are "time related". So time steps must be well evident, but for example in fig 10 and fig 11, the different time steps are just mentioned in the captions

We have added the time-steps to the figures.

>Fig 2 should be arranged in one column to avoid confusion with the further left/right side discussion and to better show the propagation of the front. >Fig 2 I can't see the stippled line mentioned in the caption.

We have emphasized the time steps more and corrected the frame.

>Fig 5b is never cited in the text even if fig 5b caption recall text for explanations.

Figure 5b is discussed on p877, from line 4 onward. We added an additional reference to the figure in section 3.1.

>Fig 5b and c are unreadable due to the tiny font dimensions >Fig 5b has 2 x axis but no dimension are present in the above axis (maybe it is time in seconds?)

Both corrected.

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>Fig 6 the dimension of porosity is % in the text and PU in the figure, please keep the same units.

Corrected.

>What fig 7 tells us more than fig 8? They look very similar to me I suggest to delete fig 7.

Figure 7 indicates the absolute numbers of pores of different volumes (i.e. the absolute frequency), whereas figure 8 indicates how the total porosity is distributed amongst pores of different volumes (the relative frequency). Both figures are discussed in the text.

>Fig. 9 I don't agree with the choice of showing a 3D figure that is for most of the people unreadable. Most of the readers (I guess) are not going to by a special glasses just to read this article. Moreover this figure is cited in the text to show interconnected pores, that are quite difficult to see. I suggest to change this in a 2D figure that show the pores before and after the dehydration with, importantly, a scale bar that is missed in this figure.

We accept the concerns and replaced the figure by a 2D version. The cube side length is given in the figure caption, as scale bars cannot be used where volumes are shown in perspective.

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Interactive comment on Solid Earth Discuss., 3, 857, 2011.