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Discussions

Interactive comment on "High temperature indentation creep tests on anhydrite – a promising first look" *by* D. Dorner et al.

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We greatly appreciate the constructive and thoughtful comments by both referees. Indeed, we are fully in line with almost all the points, which closely correspond to what we have learnt from this set of experimental results and what we intend to improve or work out in future studies. The major concerns presented in the interactive comments are twofold:

Sandra Piazolo primarily stresses the need of more detailed microstructural analysis (in particular CPO and SPO). We fully agree with this view. The reason not to go farther in this respect is that (1) we now (based on our present experience) consider the originally chosen starting material as not ideal for its microstructural heterogeneity and presence of second phase particles, (2) the chosen uniform section plane should

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have been better parallel to XZ instead of YZ with respect to starting material, and (3) problems with stability of anhydrite in air prevent renewed preparation of surfaces without severe damage. These problems cannot be satisfactorily solved for the available set of experiments; they will be considered in a new study on different material, which will then allow for a more diagnostically conclusive microfabric analysis, beyond the present first look. The very valuable suggestions are highly appreciated and will definitely find their way into forthcoming experimental strategy.

Jean-Pierre Gratier in particular addresses the applicability to natural rock deformation. He is absolutely right with that. In our paper we did not sufficiently stress that our experimental study used anhydrite primarily as a model material, and not motivated by the true need of a flow law for dry anhydrite at atmospheric pressure for geological application. We intended to point out, however, that the generally accepted weakness of evaporite horizons is not based on intrinsic properties of anhydrite in the absence of water, and that reported anhydrite CPO and SPO in natural rocks deformed at low to moderate temperature cannot necessarily be expected to be a result of dislocation creep (allowing to infer activated glide systems). As such, we are fully in line with Jean-Pierre Gratiers view that CPO and SPO can develop by anisotropic growth and dissolution precipitation creep, which was probably the case also for the geological history of our starting material.

In the following we address the points made by both referees, starting with the comments by Sandra Piazolo.

What we present in our paper is indeed a first look on feasibility and potential of the experimental approach, when applied to rock-forming minerals. Here, emphasis is on mechanical data, consistency with microstructural observations, and geometry of deformation, and not on details of microstructural evolution. This is simply prevented by the properties of our starting material, though they come surely close to what is commonplace in natural rock deformation. Nevertheless, we feel that the validity of our conclusions is not challenged by lack of more details on microfabrics. This will be the subject of a forthcoming paper. We think that with the preparation strategy chosen for the present set of experiments, some prerequisites for more in depth analysis are not met to the extent to warrant further efforts. But we have learnt our lesson. Notwithstanding, we will follow the suggestions as far as no new experiments and delicate thin sections are required, feeling that all what can be reported from the existing experiments is presented in a way that the reader can critically evaluate the results. We will include an additional figure with microstructural details in the manuscript to complement and illustrate the already presented information. Based on our experience discussed here as a ’first look’, we prefer to reserve further efforts to forthcoming experiments with more appropriate properties of starting material and modified preparation strategy.

First, we fully agree that choice of sample orientation could indeed affect the mechanical data recorded, but this effect is not expected to be pronounced for the following reasons. Despite a considerable CPO of the starting material, the scatter of orientation of individual crystals is large enough as to provide crystals with a wide spectrum of Schmid factors at the onset of deformation, which in turn would create an inhomogeneous stress field, which is characteristic for indentation tests anyway. Second, reorientation due to dislocation glide is reflected by the microfabrics of the high-strain zones flanking the neutral cone (Fig. 10 in our discussion paper), which - when viewed in our standard section plane - is probably enhancing CPO in the starting material due to tangential extension (Fig. 12 in our discussion paper). For a detailed analysis of this effect, an XZ-section parallel to the stretching lineation of our starting material would be required, or a section perpendicular to the indenter axis. Unfortunately preparation of new thin sections would require new experiments to be performed, which we intend to be reserved for more appropriate starting material. The technical feasibility of producing new sections from the remaining counterpart of available samples, with a quarter of the indenter in place, is considered not to be prospective, since (1) the indenter is normally broken out of the counterpart during preparation of the existing thin section, and (2) the saw cut has a minimum thickness of 0.4 mm, that means 20 % of

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the indenter diameter, and was placed in a way to allow some grinding to produce the available approximately median standard thin section parallel to the YZ-plane of the starting material. As such, even if preparation would be successful, the new section plane would not represent a median section through the indenter and thus be oblique to tangential extension. Thin sections normal to the indenter axis are not available and cannot be produced from available experiments. As such, the questions concerning the influence of CPO of starting material on mechanical properties and modification of CPO in the high strain zones remain a task for a new set of experiments, with different strategy of preparation. Here we must admit that we could well have thought about that before, but hindsight is always easier than foresight. Finally, we believe that the achievement of quasi steady state creep in conjunction with the (despite CPO) still considerable scatter in crystal orientation in starting material and the complex strain field can serve as a reasonable argument for principal validity of the mechanical data. Clearly, the effect of CPO should be explored in future experiments, along with details of CPO development in high strain zones based on more appropriate section planes.

The choice of the YZ plane with respect to original CPO and SPO of the starting material as the uniform standard section plane was indeed motivated by somewhat less anisometric grain shape in this plane, promising to make grain shape modification during indentation more obvious. Later this choice turned out to be unfavourable. At the time of decision we did not take into account the importance of tangential extension around the passive cone, leading to local CPO by dislocation creep, which could be in line with that of the starting material and therefore not readily isolated. This will be changed for future sets of experiments deliberately designed to address microfabrics in more detail, preferably in conjunction with numerical simulations.

We do not expect that the angle of the shear plane (apex angle of passive cone) should change with changing material properties at changing temperature. It is probably controlled by stress field rather than by material properties. The impression, understandably gained from the microphotographs, that the geometry of the passive cone may be somewhat different for different experiments is probably a consequence of variable slight deviation of the section plane from a true median cut. According to principles of conic section, only a precise median cut containing the apex of the cone provides a triangular section showing the true apex angle. With increasing shift of the section plane from the apex, the section assumes the shape of a hyperbola with increasing apparent angle between the limbs. This evolution in apparent shape is sensitive to position, which cannot be perfectly controlled during preparation.

With respect to mechanical twinning we will show a detail on the additional new figure. Thin lamellar mechanical twins are widespread in the starting material, both sets being particularly obvious in grains with maximum interference colors, cut perpendicular to [001] (cf. Fig. 1). By the way, unfortunately we have detected a mistake in Fig. 3 of our discussion paper, where [010] is to be replaced by [001]. In contrast, we observe almost no twin lamellae in the high strain zones with dynamic recrystallization to smaller grain size. In these high strain zones, which mantle the passive cone, sparse single lamellae could well be inherited in parts of grains not swept by a migrating high angle grain boundary. As such, mechanical twinning is typically absent in the high strain zones, despite stress concentration in front of the indenter edge and progressive rotational deformation within a changing stress field, which increases the likelihood for twinning at some stage of most appropriate orientation with respect to stress. Finally, twins in these high strain zones should be readily detected, as the CPO is enhanced by tangential extension, resulting in [001] preferentially oriented about normal to the section plane. This is the orientation where twin lamellae appear sharp in optical microscope. As such, our conclusion that mechanical twinning is not important or even entirely absent during the indentation creep tests at the chosen conditions, and that therefore the critical shear stress is not reached, perhaps apart from occasional local stress concentration, appears robust. Here we do not see the need for more efforts to underline the lack of mechanical twins in high strain zones, but will demonstrate the conspicuous absence using the additional new figure with microstructural details of the high strain zones.

We fully agree that the mechanical data obtained in the Müller et al. (1981) experiments cannot be directly compared with our results. Probably our message was not clear enough and could be misunderstood. What we intended to say is that the widespread idea of low strength of anhydrite rocks compared to other rock-forming minerals, as may for instance be concluded from flow law compilations in Schmid (1982) or Suppe (1985), is not based on intrinsic properties of anhydrite in the dislocation creep regime. Presence of water is obviously prerequisite, and as such deformation in experiment by Müller et al. (1981) as well as in nature may not be dominated by crystal plastic processes, but instead by more complex dissolution and precipitation (see Gratier et al., 2013 for comprehensive review). Clearly we will clarify this point in our revised manuscript. Consequently, this will also hold true for the inference on activated glide systems from natural CPO, which relies on the assumption of dislocation creep. If other mechanisms control deformation at moderate to low temperatures, CPO does not reflect glide systems. We are also fully concordant with the respective remarks by Jean-Pierre Gratier and will make this point a little more explicit in the revised version of our manuscript.

We agree that the experiments by Zaafarani et al. (2006) impressively demonstrate the potential of a comparison between 3D EBSD mapping of indents and predictions based on numerical simulations. To do justice to our approach, one must take into account that the Zaafarani et al. experiments are conducted with different goals and grossly differing conditions. Apart from the contrast in scale, plastic nanoindentation of a single crystal with specified crystallographic orientation is quite different to handle compared to a long-term creep experiment on polycrystalline material with heterogeneous microstructure, some CPO, minor second phase particles, and a two-millimeter diameter cylindrical indenter. Clearly, the Zaafarani et al. experiments show the future direction. We think that - for creep experiments on larger length scale - the first prerequisite to justify any effort in terms of comparison between experimental results and numerical simulation is the use of homogeneous material with fine grain size and devoid of significant CPO. The serial sectioning (pseudo-)3D EBSD analysis would then

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be an excellent approach. While FIB is only applicable to nanoindentation, we instead conceive a set of serial sections normal to the indenter axis, produced by successive grinding of about 0.1 mm, polishing, and EBSD mapping. This will be a very time-consuming task, which would be only warranted for a more ideal starting material as used in our present study. For such material, the microstructural evolution and CPO development along the neutral cone bounding shear zone would be of central interest. Interpretation would require a grain size between about 0.01 and 0.03 mm, however, to provide a meaningful CPO for sufficiently small areas in the complex strain field, not strongly affected by the orientation of single crystals. Whether strain is sufficient to result in CPO patterns to be correlated with local deformation history remains to be explored. Anyway, starting material with appropriate properties for the investigated length scale is of paramount importance.

In the following, we address the points made by Jean-Pierre Gratier, which made us aware of some shortcomings in the clearness of our reasoning. In fact, we very much appreciate his view and words of advice.

The probably most important issue is the motivation to investigate the mechanical behavior of dry anhydrite at high temperatures, atmospheric pressure, and low stress. Jean-Pierre Gratier is entirely right, because we missed to make sufficiently clear that, in the first place, anhydrite provides a model system in our study to explore experimental feasibility using a rock-forming mineral. In the second place, the obtained flow law for dislocation creep of dry anhydrite is extrapolated in the usual way to natural strain rates, in order to demonstrate that anhydrite is not intrinsically weak. We are fully in line with the view emphasizing the role of water in previous experimental studies as well as in weak natural detachment horizons. In the presence of water, obviously dissolution precipitation creep can take over and render anhydrite very weak at low temperatures. We fully agree with Jean-Pierre Gratier that our results are neither useful to predict rheological behavior of anhydrite in the upper crust, nor in context with geotechnical questions. One point remains, however. If dislocation creep is not responsible for CPO

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and SPO observed in evaporites deformed at diagenetic to low grade metamorphic conditions, activated glide systems operating at natural conditions cannot be derived from the respective CPO patterns. These should then be created by anisotropic growth during deformation by dissoluion precipitation creep, as suspected for other systems as well (e.g. Wassmann and Stöckhert, 2013). The role of water in dislocation creep of anhydrite is not yet explored.

On principle, anhydrite rocks are expected to persist to high grade metamorphic conditions and could well be observed in granulite facies terrains. Unfortunately we are not aware of a structural study of evaporites at such conditions, as pointed out in our discussion paper. If such an occurrence would be identified, the predicted high strength of dry anhydrite could be tested based on apparent viscosity contrasts with other minerals and rock types.

Another important question concerns the moat surrounding the indent and the lack of a complementary bulge, with other words where the material displaced by the indenter has gone. Based on microscopic examination, initial porosity is estimated as well below 1 %. A problem for microscopic inspection of porosity is the presence and inhomogeneous distribution of other minerals in subordinate amounts. Polished thin sections examined by SEM seem to occasionally show artefacts created by breakout of particles, which should not be mistaken for pores. Tiny fluid inclusions appear in some anhydrite grains, again making up a very low percentage by volume. An independent control by precise density measurements is hindered by the presence of other minerals. Obviously, given initial porosities well below 1 %, any contribution of compaction to strain achieved in indentation must be negligible. It is therefore assumed that the volume displaced by the indenter is compensated by distributed dilation on the sample scale, which cannot be precisely measured. The ratio of displaced volume to total sample volume is typically about 1.3 x 10-3. Assuming extension parallel to long sides of the sample, the length change would amount to less than 0.01 mm. We will include this information into the revised manuscript.

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Finally, we will include some remarks on disadvantages of our experimental setup, as proposed by both referees, and also include some more information on indentation techniques applied to geological materials in general. We are grateful for the respective hints.

We are ready to improve the discussion paper taking into account all considerations outlined above and questions posed by the referees in their comments, which are of great value to clarify our ideas. We plan to undertake a more in depth investigation on the evolution of microstructures and fabrics, which requires a modified preparation strategy and therefore new experiments, also on different starting material. We feel that the concept and data presented, as well as discussion and conclusions drawn in the present paper, are sufficiently robust and worth to be presented as a first look, leaving further details to be investigated in subsequent experiments.

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