Orléans, the 07th March 2025

Dear Editor, Dear Authors,

I have read in detail the manuscript entitled “Dislocation creep and glide in experimentally deformed glaucophane aggregates” by Lonnie J. Hufford and others. This works aims at constraining, on the basis of 5 deformation experiments, both the microscopic deformation processes and the corresponding macroscopic flow laws. This works is integrated in a larger series of works dedicated to the rheology of the various rocks deforming along the subduction interface.

While rheological contrasts are clearly visible in deep metamorphic rocks from subduction zones, consistent rheological flaw laws are missing, and this work is very relevant in this respect. A difficulty lies in the fact that part of the deformation processes described in this work pertain to the transition between fully plastic deformation and brittle/frictional deformation. For such conditions, the different possible processes operating are multiple (involving fracturing, frictional slip, chemical mass transfer along grain boundaries, plasticity etc…) and very careful observations are necessary.

The present work falls short of such necessary observations, as the documentation of the microstructures is really poor (see full comment below). For example, none of the microstructures invoked (subgrain boundaries, lobate grain boundaries) are well documented, while the SEM pictures are reminiscent of brittle deformation, which is never considered here.

The experimental dataset is also very short: 5 deformation experiments, without any replicate, to characterize 2 different types of deformation mechanisms and cover ranges of variations in temperature and in strain rate. It includes one deformation experiment where the grain size is different from the rest, and the authors, instead of repeating the experiment with the proper grain size, chose to keep it but to exclude the mechanical data from the dataset used to derive the flow laws. The microstructural work is somehow questionable for load-stepping experiments covering two deformation regimes, as it is then impossible to tie microstructures to either one of the deformation regime. So further experiments are necessary, including a few replicates of the experiments shown, constant strain-rate experiments in either one of the regimes to analyze the microstructures, a new “700°C” experiment etc…

The lack of balance between the observations and the interpretation is apparent in the structure of the work: 40 lines and 4 figures of results, to fully describe a complex mechanical behavior, vs. 140 lines and 4 figures for the discussion. A companion paper is evoked, what is hard to understand, given the fact that the present work is missing many data and observations.

As a result, my conclusion is that the work is an interesting, promising experimental work, on an interesting topic, but it is still far from being completed. I suggest to add the missing data (additional experiments, additional observations, all necessary) and to resubmit the work. If a companion paper is submitted in parallel, an idea to consider (I cannot be sure of it because I have had no access to it) might be to merge the two manuscripts.

Best Regards

Hugues Raimbourg

PS: 18th of March – The editor sent me the link to the companion paper. I checked it quickly, and it seems that there are indeed many data and observations that are relevant to the present manuscript. I do not find it relevant to split the work into two distinct manuscripts, because the result is that the present paper lacks the necessary information to stand on its own.

Plus, I found that some part of the text was repeated between the companion paper and the present one. The last part of the companion paper (L420-437) and of the present manuscript (L271-287) are identical but for a few words. That is an additional argument for merging the two papers. But even more, I consider it an ethical and professional misconduct to copy-paste whole sections of the Discussion between distinct papers.

Main comments

1) Microstructures

There is much microstructural characterization that is missing:

-compositional mapping to show whether dissolution-precipitation, or reactions, or any chemical process occurred and played a role in deformation

- EBSD data: Distribution of misorientation angle of low angle/subgrain boundaries are necessary, as they are directly connected to the active slip system. Pole figures should be shown as well, even if they might not be very informative, because the low amount of strain prevents a clear CPO to develop. Grain size distribution

-Comprehensive set of SEM observations, showing the grain boundary geometries (and if present, the lobate GB), the cracks, the grain size etc…

-pictures showing contrasts in deformation microstructures as a function of temperature (using EBSD, SEM, optical microscopy…)

-TEM work on internal defects

The figures that are provided are too few and of too little quality: In Fig. 2, the optical microscopy images should be zoomed to highlight the microstructures to be observed (and please use the same scale bar on all comparable pictures, for example the large views on the left). In Fig.3, the EBSD maps are obscured by horizontal, superimposed “bands”. There is no clear demonstration of the widespread occurrence of subgrains, neither lobate grain boundaries in the EBSD maps. In Fig. 4, the contrast in BSE should be increased to be able to show the presence or absence of newly precipitated amphibole.

Furthermore, a problematic shortcoming of the present work is that the microstructures that are shown reflect a combination of different stages, with potentially different deformation mechanisms. In other words, one cannot ascribe the microstructures to the low- or high-stress regime. A more proper approach would be to combine the load-stepping experiments with constant strain-rate experiments, either in low- or high-stress regime, to observe and describe the relevant microstructures and their difference.

2) Mechanical data

Strain-rate stepping experiments using powders in shear geometry: the final strain achieved is 0.8. The initial stage of compaction, simply to transform a powder with a porosity, onto a non-porous solid sample, takes some strain. For example, the very large sets of experiments on granitoids powders in Griggs shear experiments made by Matej Pec (Pec et al., 2012; Pec et al., 2016) show that strain of the order of $\gamma \sim 1.5$ is necessary to compact the powder, close the porosity and reach steady-state microstructures and mechanical behavior. So it is arguable that the present experiments relied on

steady-state microstructures, hence the bulk properties they derived are applicable to creep properties (which is implicitly a steady-state process). Furthermore, it is conceivable that the mechanical measurements performed in the present work are strain-dependent, hence cannot be converted into a flow law. This is why I suggest repeat one or several of these load-stepping experiments, and to apply, after the last, high load step, a step with a low value of load, comparable to the first step, to check that the mechanical values are not affected by strain and the associated microstructural evolution (see the approach in (Ghosh et al., 2024)). Discarding, through experimental evidence, such a strain- or history dependence is essential to support the derivation of a flow law (where by definition strain and history are absent, as steady-state behavior is assumed).

Brittle/semi brittle behavior: The low-temperature and high-strain rates applied in these experiments may lead to frictional deformation in the brittle regime (see the very large stress exponent and mechanical data in (Marti et al., 2017; Pec et al., 2016)). The fine-grained domains of amphibole in Fig. 4 might be tentatively interpreted as grain crushing, consistent with the interpretation as brittle deformation. I am not claiming that it is the case, but great care should be taken in the present work to discriminate plastic processes (such as the formation of subgrains and their evolution into independent, recrystallized grains) from brittle ones. As stated above, a much more careful microstructural work is necessary to fully support the conclusions of the manuscript.

The fit to a flow law is very disappointing for the “glide” part of the dataset. The 700°C sample shows discrepant results, i.e., a much lower strength compared to what is predicted. This is attributed to a larger initial grain size. Fine, that sort of things can happen during sample preparations. But then, why not repeat the experiment with the same powder and the same grain size as the other 3 samples, to have a robust and comparable “700°C” experiments? Instead of that, the authors discard the experiments and fit their glide law with 3, instead of 4 temperature data, and derive a law out of a very narrow range of temperature (hence large uncertainties when extrapolated to different temperatures).

Line-to-Line comments:

L84-85: ”A modified version of the open source code RIG (https://mpec.scripts.mit.edu/peclab/software/) for the ETH Zürich Griggs apparatus was used to process the mechanical data.” : There are several possible corrections to the raw data, in particular concerning the stress values (Heilbronner et al., 2015). Please provide access to a comprehensive description of the routines applied to the raw data.

L132-138: please provide the compositional maps that shows that precipitated material is limited.

L141-143: The presence of subgrains and lobate grain boundaries is really poorly documented.

L150: the interpretation of high stress exponent as dislocation glide is questionable. It might well be the result of frictional deformation in the brittle regime.

Figure 3: The quality of the map is really poor (especially the deformed sample b and d). A surprising feature is that there is hardly any clear plastic feature in the deformed sample, in spite of the large strain (up to 0.8): the inherited grains are not elongated, preferentially orientated parallel to the shear plane etc… This suggests that most of the strain is actually taken up by the compaction of the powder, which raises many questions as to the mechanical relevance of the initial, low-stress load steps.

Fig. 3: The most visible difference between hydrostatic and deformed sample is the abundance, in the hydrostatic sample, of “white”, unindexed areas. Do these correspond to porosity, or to really

unindexed amphibole grains? Finally, one would expect a low degree of internal misorientation in recrystallized domains (Cross et al., 2017). In contrast, a surprising feature of the deformed sample shown in Fig. 3d is that small grains domains, presumably formed by dynamic recrystallization, show actually larger misorientation than inherited grains.

On this figure, please use the same scale bar for hydrostatic and deformed sample.

References

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