



Interactive comment on “Artifacts from manganese reduction in rock samples prepared by focused ion beam (FIB) slicing for X-ray microspectroscopic analysis” by Dorothea S. Macholdt et al.

Anonymous Referee #2

Received and published: 15 October 2018

General comment:

The paper reports on a study investigating Mn rich rock coatings to find means to identify the different mechanisms in the genesis of the crust formation. The authors have examined the oxidation states of Mn in the crusts using FIB (focused ion beam) slicing and scanning transmission X-ray microscopy. The finding of the manuscript is that preparation of specimens by FIB and monitoring of samples by SEM cause artifacts that complicate the oxidation state analysis.

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The reader cannot avoid an impression that the manuscript is a spin-off produced by an ambitious study originally intended to resolve the profile of Mn oxidation states all the way through the varnish layer and to track the different mechanisms behind the development of the coating. However, as sample preparation techniques remains an issue within geosciences, the manuscript makes a fair contribution to this field of science.

Specific comments:

Page 8 line 32: “To verify whether a layer of modified material is actually distributed homogeneously on the surface of the sample”

Why would you assume an even distribution? Please justify.

Page 11 line 10: “As there is at this time no alternative to FIB as sample preparation technique to produce intact ultra-thin slices of rock samples,”

The reader might wonder which are the benefits provided by FIB compared to, e.g., Argon ion slicing that has been also used in production of thin foils especially for TEM. According to this statement, you don't consider Argon ion slicing as an alternative to FIB. However, if this is the case especially for the samples used in this study, the reader would appreciate some reasoning.

Page 3 line 33: “Here we report about our findings observed during the investigation of the Mn oxidation states in 14 rock varnish samples, collected in different environments and countries.” and Page 4 line 2: “For the sake of brevity, and since all samples showed the same phenomena, these findings will be exemplified using measurements on one of the samples.”

It is interesting that no differences between the varnishes were found especially as you have previously reported (Macholdt et al. 2017) that layers of Mn-rich material and structures like cavities vary significantly between coatings of rock samples collected from different environments and regions. Perhaps you could refer to your earlier study to emphasize the importance of the finding of this manuscript - that the sample prepa-

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ration of this sort produces similar kind of artifacts no matter what the structure of the varnish is.

Page 10 line 8: “we found that artifacts are produced during the preparation of the samples by FIB and monitoring by SEM, which create a high degree of uncertainty for oxidation state analyses.”

The reader would appreciate a quantitative estimate. Would it be possible to give a rough estimate on how much sample preparation of this kind adds to the total uncertainty – on the basis of the case presented in the manuscript?

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