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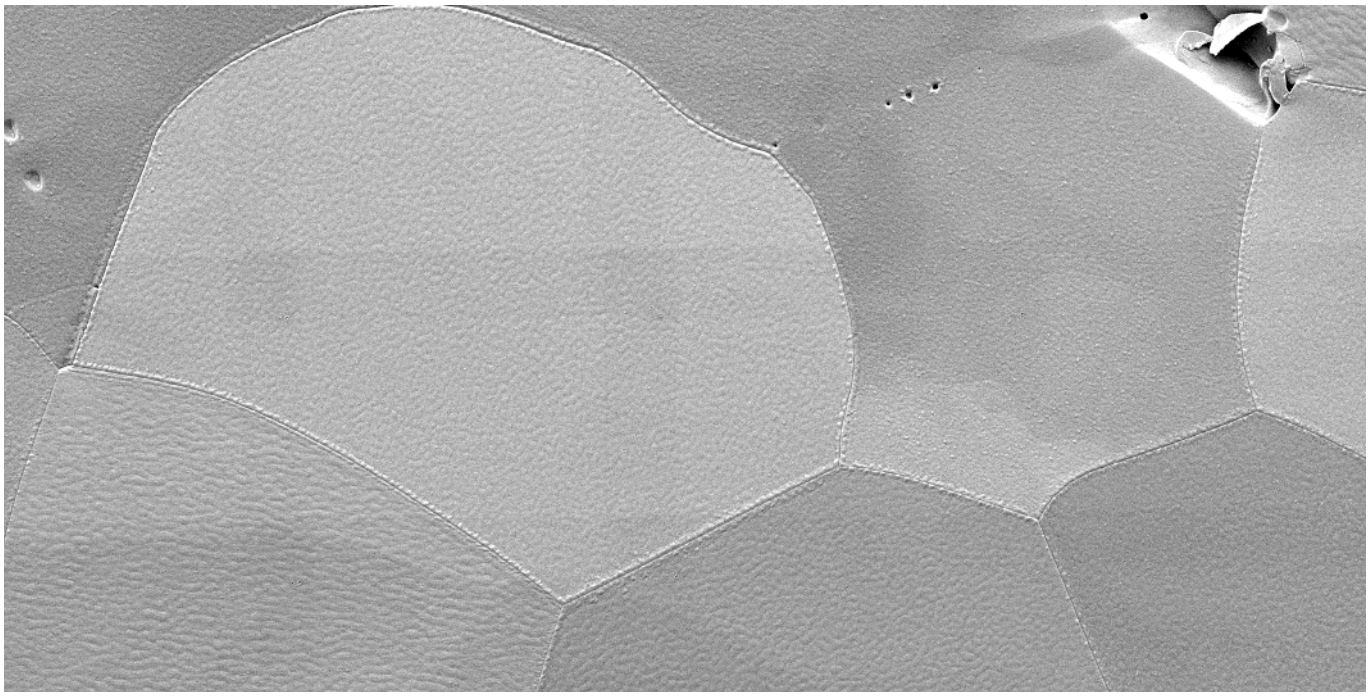
# Triple-beam Ar-Ion-Milling with a Rotary Stage to Decorate Grain Boundaries and Substructures in Rock Salt



Secondary electron micrograph of high-angle Ar-ion etched surface of polycrystalline rock salt.


## Summary

Decoration of grain boundaries in polycrystalline rocks has a long tradition in Structural Geology as in a monomineralic rock the recrystallized grain size is a good indicator for the paleostress conditions. Understanding the mechanical properties of rock salt and its deformation behavior is of major importance for the prediction of long-term stability of nuclear waste repositories, and for our understanding of the dynamics of salt-related sedimentary basins which host the majority of oil and gas deposits on Earth. Common methods to expose grain boundaries and substructures in rock salt are chemical etching procedures using brine or water or the gamma irradiation method.


## Aim

Here we present an alternative method to decorate microstructures in rock salt: Polishing and contrast enhancement using Argon Broad Ion Beam (BIB) polishing with a rotating sample stage.

## Sample material

Sample material shown is synthetic rock salt produced by cold-pressing at room temperature in an extrusion machine (from 0 to 4.5 GPa in 15 s) from a salt mush made of pure NaCl-powder (NaCl content >99.9 %, grain size <10 µm) and NaCl-saturated solution. The resulting cylinder with a diameter of 2 cm was cut into 4 mm thick cylindrical slabs using a diamond blade rock saw. These samples were stored at room temperature and at atmospheric pressure in an air tight vessel for more than 10 years.

## Results

Surface grinding produced a rough but regular surface with pronounced grooves in grinding direction. Water etching entirely removed the surface grooves. Moreover, it produced a smooth surface and exposed structures like pores and grain boundaries that could not be recognized directly after grinding. Investigating the sample at high resolution it could be speculated that even subgrain boundaries and different crystal lattice orientations could be distinguished.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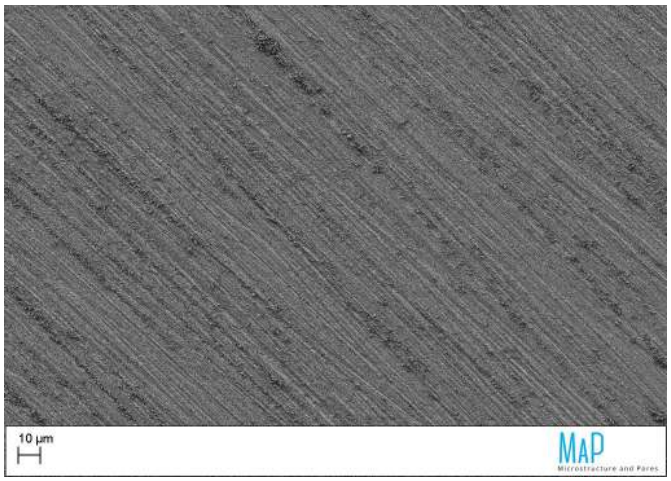

Figure 1: Surface of hand polished sample

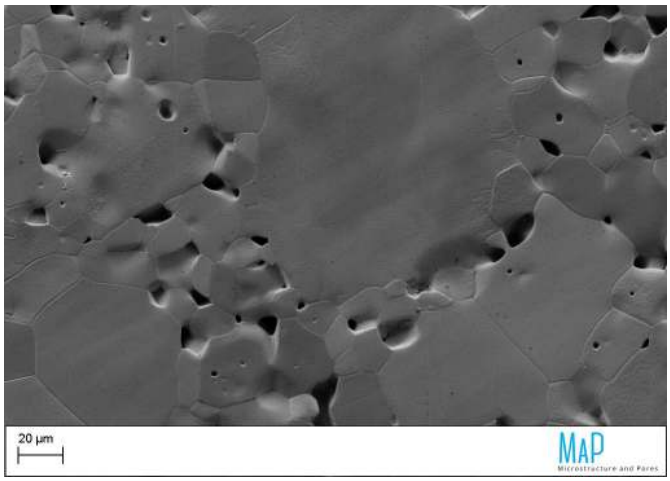

Figure 2: Surface of water-etched sample

Disadvantage of surface etching using pure $H_2O$ is that the process is difficult to control. This was visible in pronounced dissolution of e.g., pore edges. Even using the far more sophisticated etching method, established by Urai et al., 1987, with slightly undersaturated NaCl solution under controlled conditions, would be highly influenced by the presence of pores and the defect structure of the grains, and hence needs to be adjusted to the characteristics of each single type of salt sample.

BIB-polishing at low angle produced a smooth surface of the entire surface area (Ø 2 cm). SEM imaging allowed determination of the grain structure based on the density contrast caused by different mineral orientations. The grain boundaries are exposed as thin grooves and

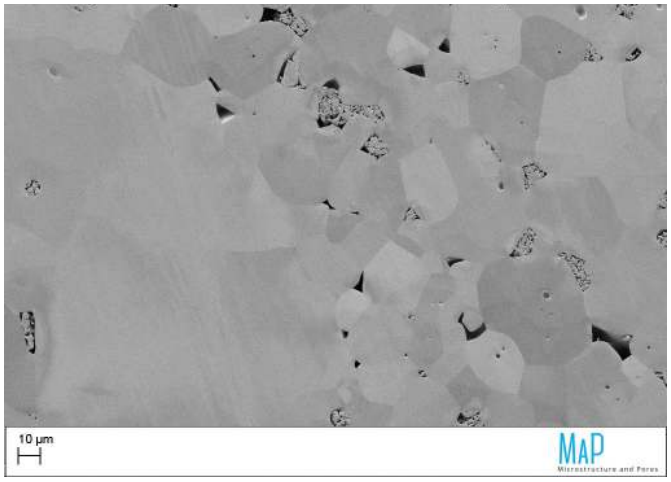

Figure 4: Surface of BIB-polished sample

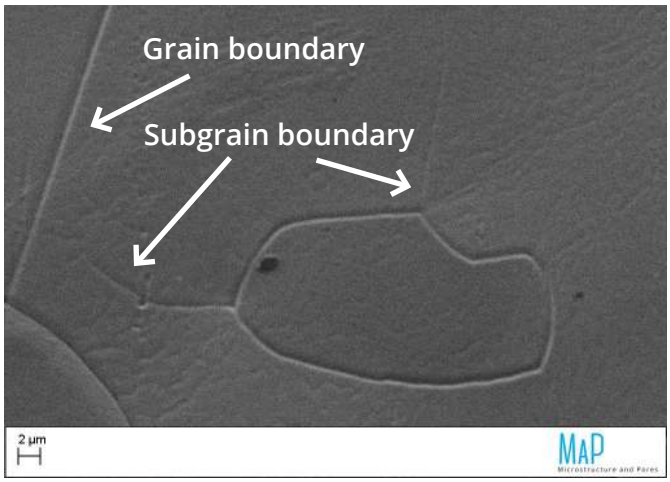

Figure 3: Grain boundaries visible after water etching

are only visible at very high magnification . The pores show sharp etches and some of them were filled with small salt grains, which were presumably grinding dust.

Grain boundary decoration was done by means of etching using the rotary stage at high angle with respect to the orientation of the triple beam (Figure 7). This treatment caused the exposure of grain boundaries as grooves that were visible also at low magnification in SEM. The bulk grains showed a distinct surface pattern that varies from grain to grain and could be interpreted to be dependent on the crystal lattice orientation. However, this assumption could only be validated employing EBSD analysis.

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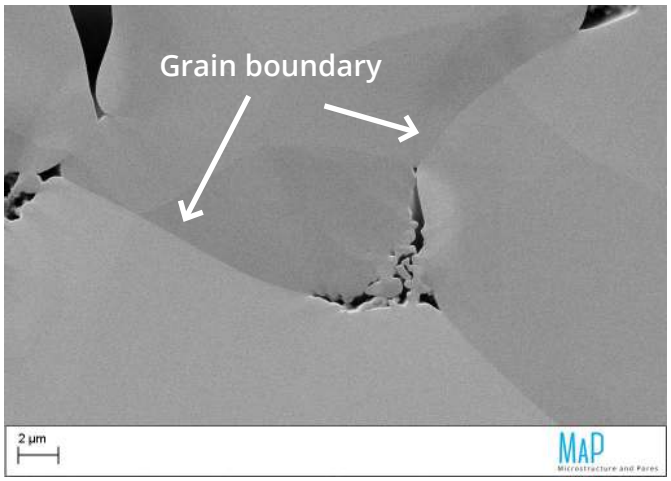

Figure 5: Low-angle Ar-ion polished rocks salt

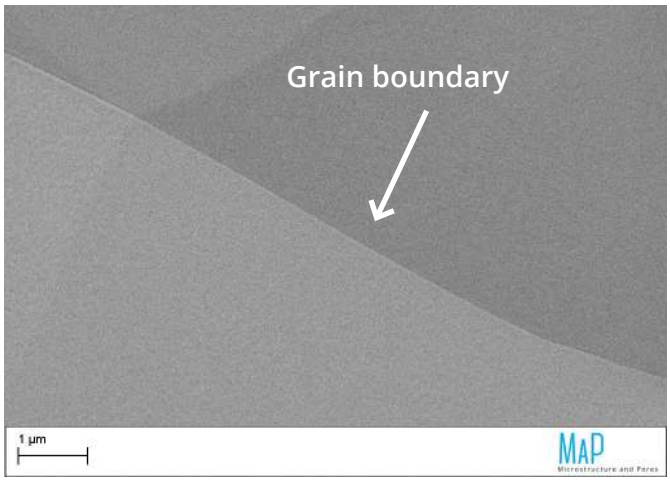

Figure 6: Detail of low-angle Ar-ion polished salt

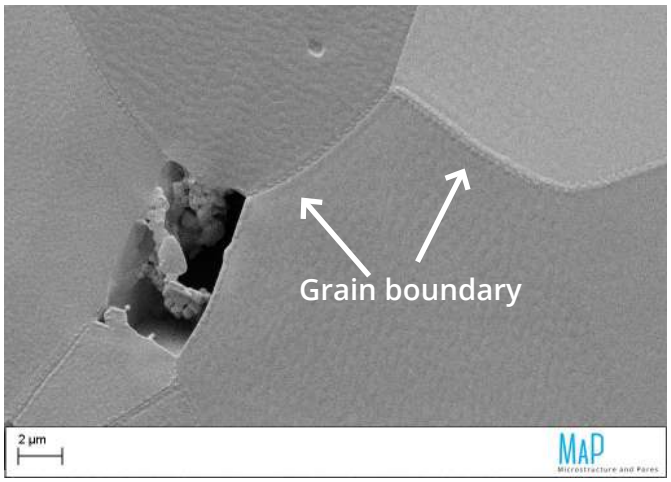

Figure 7: High-angle Ar-ion etched rocks salt

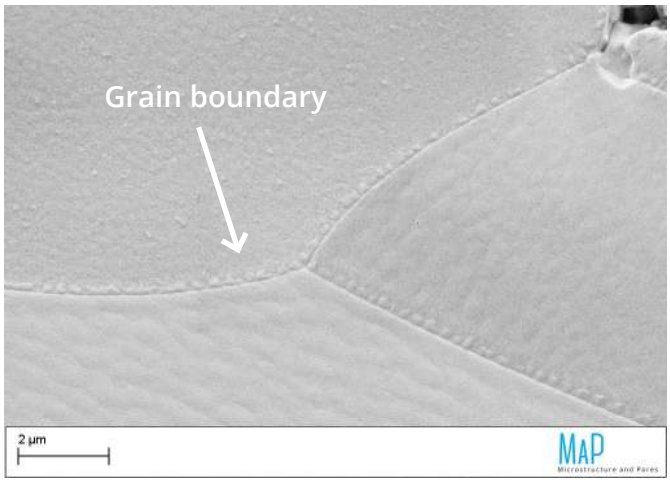

Figure 8: Detail of high-angle Ar-ion polished salt

## Outlook

Grain boundary decoration using BIB-polishing is an efficient and promising method to expose grain boundaries and substructures in polycrystalline rock salt. As the irradiation process is a mechanical process it could have advantages compared to the traditional chemical etching in terms of reproducibility and transferability to other samples and materials. The method will be evaluated and validated using complementary methods such as EBSD and/or the gamma irradiation method [1]. The applicability of the method to other monomineralic rocks such as quartzite is likely and worth to explore as these kinds of rocks are far more difficult to prepare with chemical etching.

## References

(1) Urai JL, Spiers CS, Peach CJ, Franssen RCMW, and Liezenberg JL: Deformation mechanisms operating in naturally deformed halite rocks as deduced from microstructural investigations. Geologie en Mijnbouw 66: 165–76 (1987).

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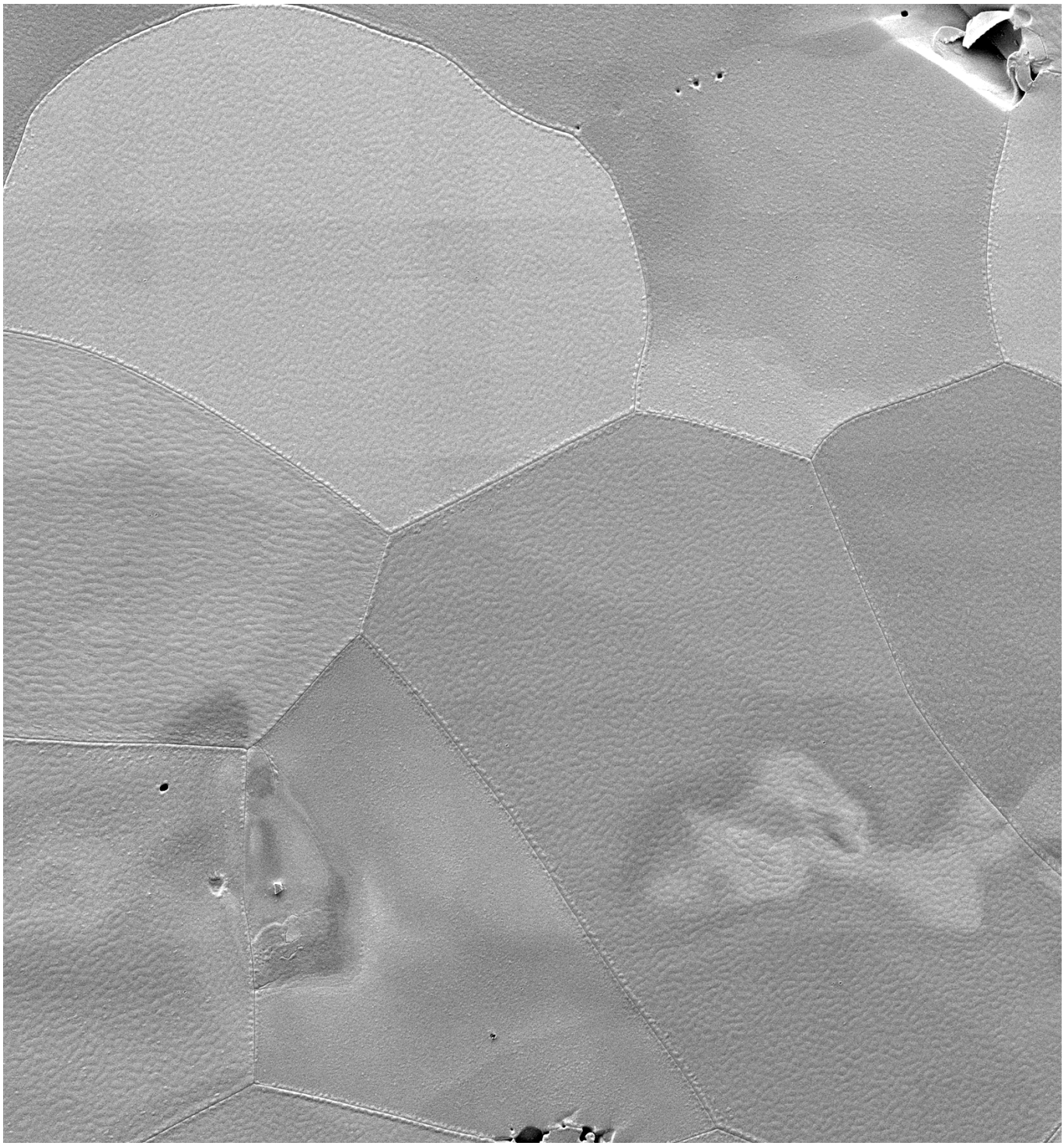



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