

CCC Cavern Closure Consortium

ZUIDWENDING WP2.3 Small-Deviatoric-Stress Creep Tests in the Mine



9 March 2024

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EXECUTIVE SUMMARY

In an abandoned cavern, brine pressure increases and becomes relatively high. Deviatoric stresses are small in the rock mass and tend to decrease at the cavern wall. Standard constitutive laws are poorly fitted to these special conditions, as the effects of low deviatoric stresses are not commonly studied at the laboratory in the 5-20 MPa range.

The constitutive law of salt at low deviatoric stresses, governed by pressure-solution creep is therefore of particular significance for the behavior of salt caverns after abandonment.

Four samples from Zuidwending were tested at small deviatoric stresses in the Altaussee mine in Austria to determine a set of parameters for the pressure solution creep law. Loads applied on each sample are given in Table 1. The samples were prepared, pre-compacted and pre-deformed at the IfG laboratory in Germany (Table 2):

- Deviatoric loading - the aim was to deform 2 samples to 20% (axial strain) while applying a 20 MPa confining pressure and a 90 °C cell temperature.
- Hydrostatic pre-consolidation – 2 samples were hydrostatically loaded at 90 °C and 20 MPa for a defined duration of ca. 80 hours.

Table 1. Loads on the samples.

Sample Name	Weight (kg)	Load (MPa)	
		Calculated on initial size	Calculated on final size
Zuidwending_1	233	0.61	NA
Zuidwending_2	233	0.61	NA
Zuidwending_3	1176 - 628	2.98 - 1.59	NA
Zuidwending_4	233	0.51	0.61

Strain rates are calculated using a weighted moving-average method. A sliding window of one week is considered. The weighting is performed using binomial coefficients. Table 2 provides the main results of the creep tests.

Table 2. Main results of the creep tests.

Sample Name	Load (MPa)	Total strain ($\times 10^{-6}$)	Final strain rate ($\times 10^{-12}$ /s)	Av. grain size (mm)	Pre-deformed at IfG
Zuidwending_1	0.61	561	2.63	0.34	
Zuidwending_2	0.61	525	2.51	0.46	
Zuidwending_3	2.98 - 1.59	1060 – 37.5	8.00- 1.55	0.61	
Zuidwending_4	0.61	1128	5.00	0.30	×

Figure 1 shows the results of the Zuidwending salt tests in comparison with other measurements in the literature. It should be borne in mind that the tests were carried out at a temperature of around 8°C and that therefore the creep rate would be greater at the temperature of the salt at the depth of the caverns, which can be estimated to be around 30 to 40 °C. Grain size was measured from reflected light micrographs after completion of the mine tests.

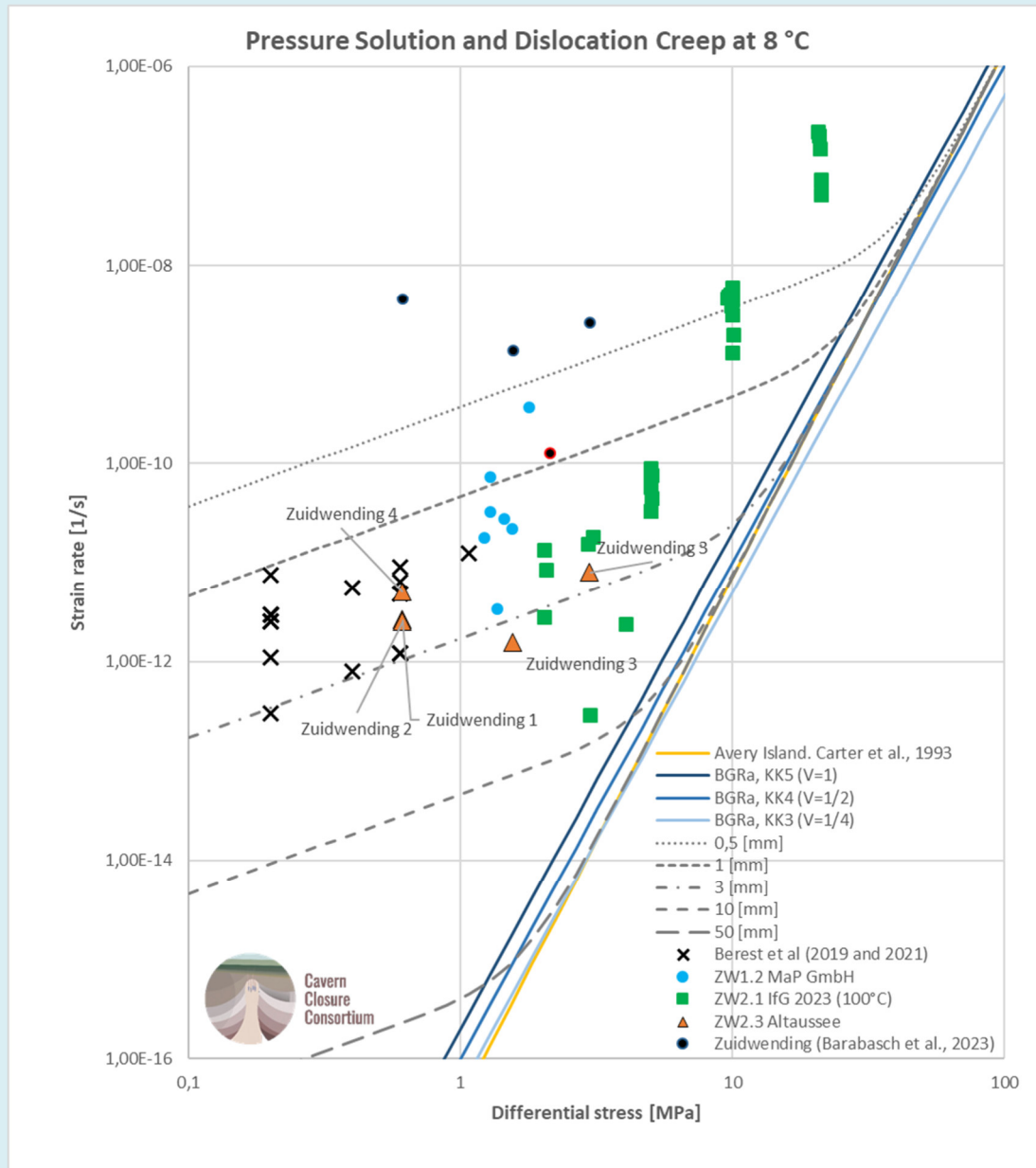


Figure 1. Comparison with other salts in the literature (Zuidwending=orange triangles).

Pressure solution creep can be described by equation (1)

$$\dot{\epsilon}_{ps} = \frac{A_{ps}}{TD^3} \exp\left(-\frac{Q_{ps}}{RT}\right) \sigma \quad (1)$$

where A_{ps} is a prefactor, Q_{ps} is activation energy and D is average grain size.

When a $Q = 24.2$ kJ/mol is considered, the following set of parameters can be back calculated from the tests on the non-pre-deformed samples 1 and 2:

$$\begin{cases} A_{ps} = 250 - 414 \text{ K} \cdot \text{mm}^3 / \text{MPa} \cdot \text{yr} \\ D = 0.7 \text{ mm} \\ Q_{ps} / R = 2910 \text{ K} \end{cases}$$

We compared the microstructure of the salt core pieces before and after deformation in the mine using a range of microanalytical methods. No microstructural information is available after completion of IfG pre-deformation prior to mine testing. The preparation of a set of sister samples was not included in the original design of the study. When evaluating the change in microstructure after completion of the mine test in the pre-deformed specimens, it should be borne in mind that most of the changes in microstructure are likely to be related to the creep deformation in the IfG rather than the deformation in the mine.

While the overall microstructure of the tectonically deformed samples and the non-pre-deformed mine samples was very similar, the pre-deformed showed newly formed, often cubic-shaped grains in thin section, replacing matrix grains with higher degree of stored energy, which is generally shown by the presence of subgrains or dense slip bands.

The grain size in all creep-tested samples diminished, differing by a factor 3.1 of for sample Zuidwending 1, a factor of 2.3 for sample Zuidwending 2, a factor of 1.7 for sample Zuidwending 3, and a factor a 3.5 for the pre-tested sample Zuidwending 4 (Table 2).

The grain boundary structure undergoes visible changes when comparing the initial twins with the creep-deformed samples. While grain boundaries in the initial samples typically exhibit smoothness, creep-tested samples often display serrated grain boundaries. Serrated grain boundaries tend to form perpendicular to the orientation of the maximum principal stress of the creep test, suggesting dissolution due to increased contact stress. Moreover, most grain boundaries in the creep-tested samples show efflorescence with NaCl precipitates along the grain boundary visible after thin section preparation. This is believed to represent in-situ grain boundary fluids reaching the sample surface due to capillary forces arising from sample preparation (Schenk and Urai, 2004; Desbois et al., 2012). Efflorescence is seldom observed in thin sections of the initial samples. Additionally, the structures of the observed precipitates at the interface of adjacent grains, as revealed by SEM, suggest the occurrence of successive generations of precipitates reminiscent of syntaxial vein growth. The structures formed on the grain surfaces vary from amorphous to fibrous to blocky crystal shapes, occasionally accompanied by porosity. These observed structures may imply a process of grain boundary opening, facilitating the precipitation of NaCl depending on the concentration gradient in a dissolution-precipitation process (Urai and Spiers, 2007, and references therein). Furthermore, locations with grain overgrowths at grain triple junctions are occasionally visible. A notable contrast is observed between the grain boundary surfaces of creep-tested samples and the initial ones, with significantly fewer imprints of regular fluid inclusion arrays on the grain surfaces of the creep-tested specimens.

Overall, the mine test results are in good agreement with the earlier mine test results, and with the predictions for pressure solution creep. Almost identical microstructures of the Zuidwending 1 and 2 samples resulting in almost identical creep rates underline the accuracy of the creep tests performed under stable conditions in the Altaussee mine. Finally, the pre-deformed samples have finer grain size and deform faster than the untreated samples, but not as much as expected from the grain size difference. In the pre-deformed samples, both of which creep slower than predicted, we noticed that the small new grains appear to be depleted in the grain boundary fluid visible in the microscope, which could be an explanation that they therefore have less potential to contribute to pressure solution creep (Urai et al., 1986; Watanabe and Peach, 2002). Further investigation and especially comparison with other mine test results would be required to conclude on this.

NOMENCLATURE

Latin symbols

SYMBOL	DESCRIPTION	UNITS
A_{dc}	Pre-factor for dislocation creep	/MPa ⁿ -yr
A_{ps}	Pre-factor for pressure solution creep	K.mm ³ /MPa-yr
D	Average grain size	mm
d	Sample diameter	mm
h	Sample height	mm
n	Exponent of dislocation creep law	—
Q_{dc}	Activation energy for dislocation creep	J/mole
Q_{ps}	Activation energy for pressure solution creep	J/mole
R	Universal gas constant	J/mole-K
T	Absolute temperature	K
u_i	Measured displacement	μm

Greek symbols

SYMBOL	DESCRIPTION	UNITS
ε	Strain	—
$\dot{\varepsilon}$	Strain rate	/s
σ	Deviatoric stress	MPa

1. INTRODUCTION

1.1 Background

In an abandoned cavern, brine pressure increases and becomes relatively high. Deviatoric stresses are small in the rock mass and tend to decrease at the cavern wall. Standard constitutive laws are poorly fitted to these special conditions, as the effects of low deviatoric stresses are not commonly studied at the laboratory in the 10 °C-50 °C range.

In 2014-2016, an experimental program (Bérest et al., 2017, 2019, 2022), supported by the Solution Mining Research Institute (SMRI), proved that, in the 0.2–1 MPa deviatoric stress range, creep rate of natural salt sample was much faster – by several orders of magnitude – than the creep rate extrapolated from what is currently observed during standard creep tests performed in the 5 – 20 MPa range at the laboratory (Figure 2). Microphysical modelling and triaxial deformation data have demonstrated since about 1983 that pressure-solution, a mechanism in which grain size and brine content play an eminent role, is especially active in the small stress domain (Urai et al., 1986; Spiers et al., 1990).

The constitutive law of salt at low deviatoric stresses, governed by pressure-solution creep is therefore of particular significance for the behavior of deep caverns after abandonment.

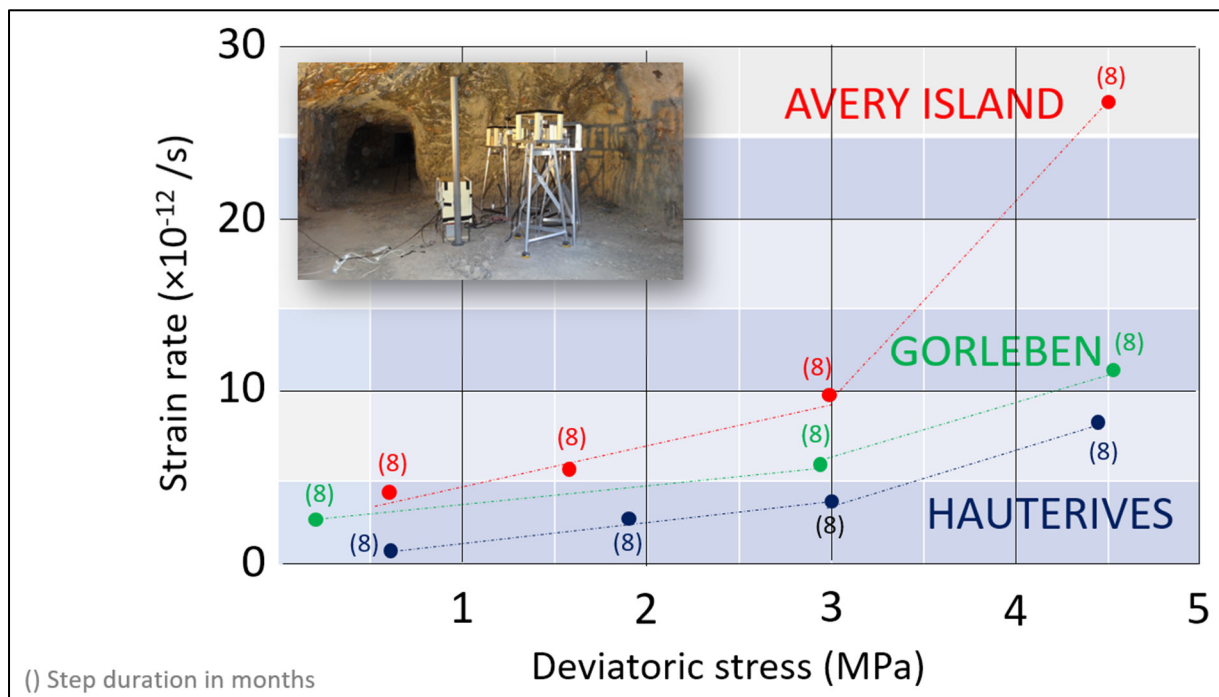


Figure 2. Example results from creep tests performed at the Altaussee mine.

1.2 Scope of work

Four samples from Zuidwending were tested at small deviatoric stresses in the Altaussee mine in Austria to determine a set of parameters for the pressure solution creep law. Two of the samples were pre-deformed at the IfG laboratory in Germany (see WP2.2).

1.3 Methodology

1.3.1 Temperature and humidity fluctuations

When performing accurate creep tests, temperature and hygrometry fluctuations are by far the main concern. Both have a strong influence on creep rates. In addition, even small temperature changes generate thermal strains which can be larger than cumulated creep strain during a creep test. For this reason, tests are performed in a remote gallery of the Altaussee Salt Mine in Austria where long-term changes in temperature and relative humidity are smaller than 0.01 °C/yr and 0.5 %RH, respectively.

1.3.2 Applied stress

During a creep test, fluctuations in the applied stress also are a concern, that's why dead weights are used. A cylindrical salt sample, $d = 70$ mm, $h = 140$ mm, is set between two horizontal duralumin plates in the upper part of the testing device. Dead weights are set on a platen in the lower part of a mobile rigid frame. The frame weight is transmitted to the upper duralumin plate through a small metallic ball (Figure 3).

The range of stresses that could be applied to a sample with such a system is 0.02 to 1 MPa. "Filling the gap" between 1 and 5 MPa stresses implied applying larger stresses. In fact, the platen is not able to accommodate many steel disks (the dead weight), therefore three of the creep devices are equipped with a « force-multiplier »; i.e., a cantilever system which allows applying stresses up to 5 MPa (Figure 5). It was checked that the additional weight added to the platen was multiplied by a factor of 5.27 when 25 kg disks were used and 5.43 when 10 kg disks were used.

1.3.3 Measurement of axial displacements

Creep rate can be computed by comparing strains $\varepsilon_1 = \delta h_1 / h$ and $\varepsilon_2 = \delta h_2 / h$ measured at two different times, t_1 and t_2 , or $\dot{\varepsilon} = (\varepsilon_2 - \varepsilon_1) / (t_2 - t_1)$. When, for example, $t_2 - t_1 = 10^5$ s (one day) and $\dot{\varepsilon} = 10^{-12}$ s⁻¹, then $\varepsilon_2 - \varepsilon_1 = 10^{-7}$. Because the sample height of the specimen tested for this study is $h = 140$ mm, axial displacements must be measured with a very good accuracy. Special sensors (*Solartron* linear encoders) were used; their resolution is 1/80 μm.

The lower duralumin plate is fixed. A purely rigid movement (rotation + vertical displacement) of the stiff upper plate can be expected. To detect such movements, three sensors are needed. Because the tests are 2-year long, it is essential to assess the possible drift of the sensors, which must not be confused with the actual creep of the sample. For this reason, four high-resolution displacement sensors are positioned in two vertical planes at 90° angle for redundancy (Figure 4). In fact, the four displacement-vs-time curves (u_1 , u_2 , u_3 and u_4) do not overlap. However, the two curves, $(u_2 + u_4)$ and $(u_1 + u_3)/2$, almost perfectly overlap (see Figure 12 p°18 for instance), which proves that, in addition to the average vertical displacement of the upper duralumin plate, a rotation of this plate takes place: the four displacements measured for one sample are consistent, and there is no or small drift of sensors. To be precise, the drift was measured to be 0.3 μm/yr.

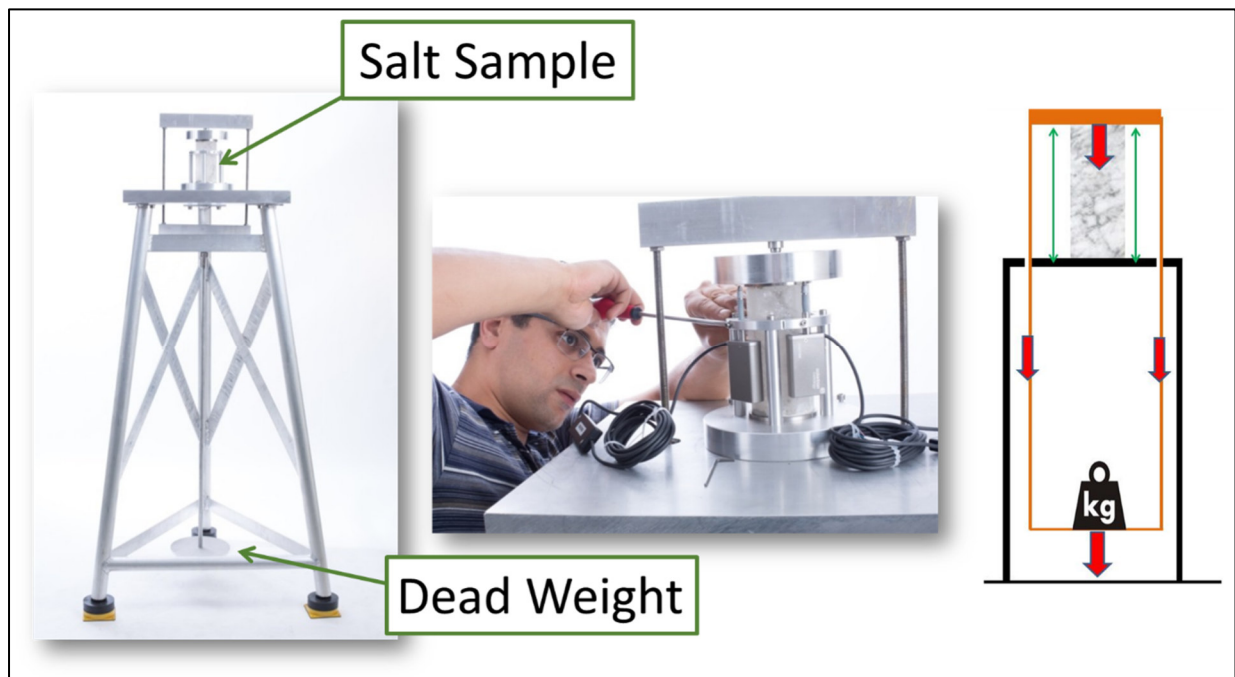


Figure 3. Dead weights are used for the loading.

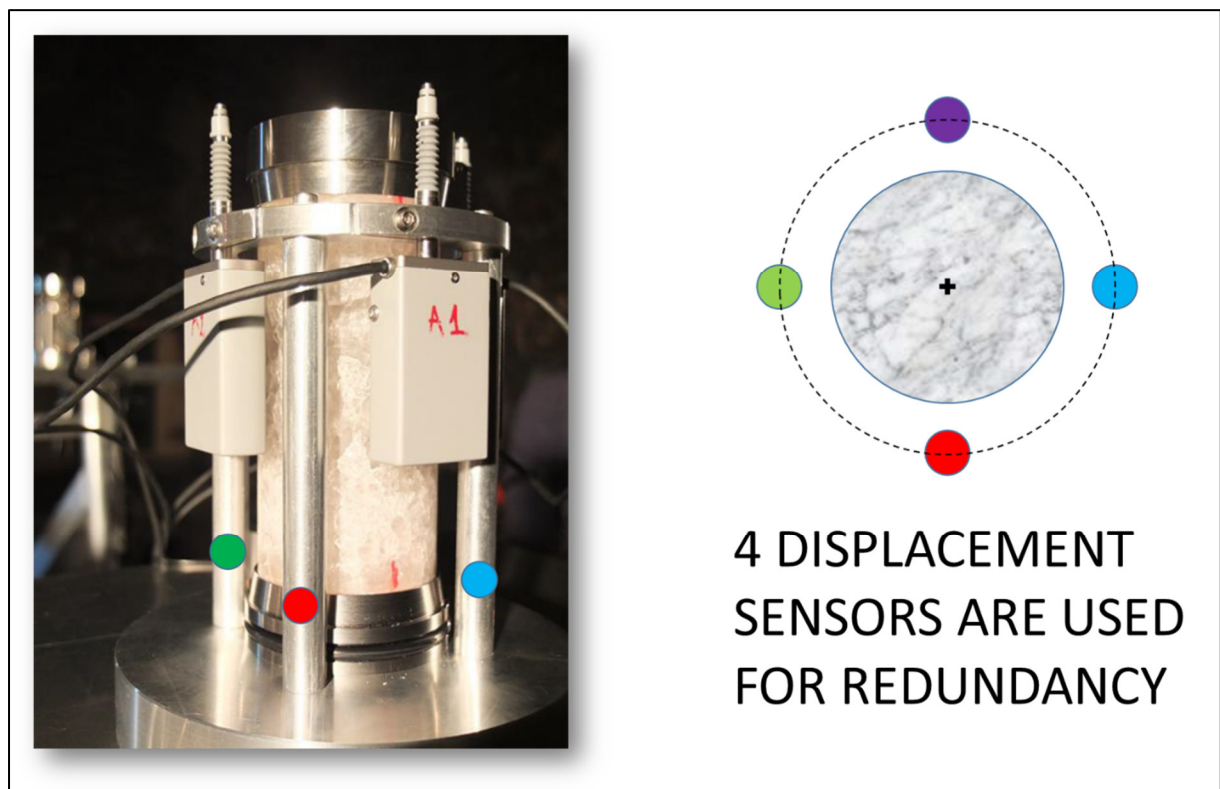


Figure 4. Four displacement sensors are set around each sample.

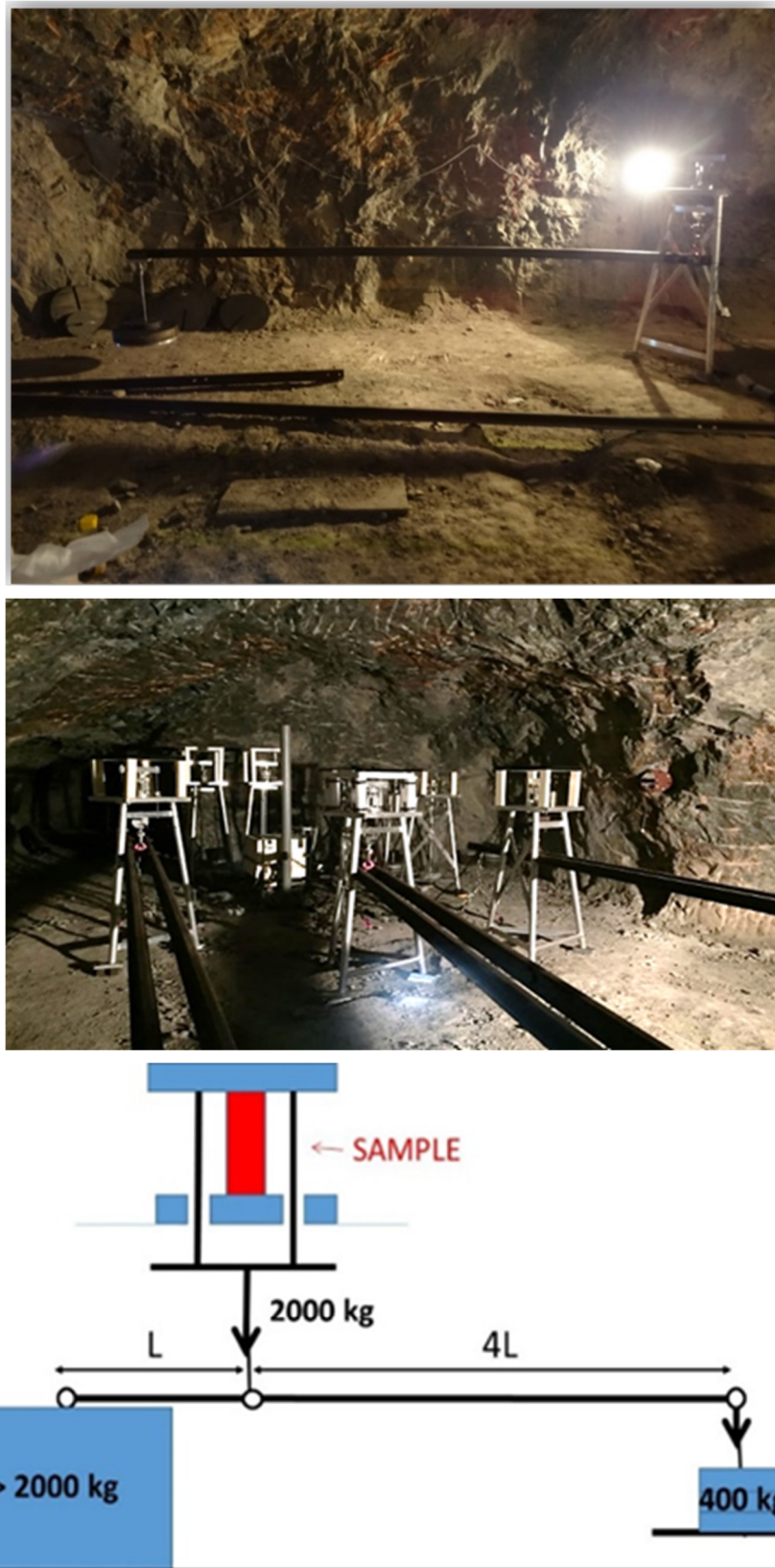


Figure 5. A cantilever beam (above), the three cantilever systems in the drift (middle), principle of the force multiplier (bottom).

1.3.4 Methods of microstructural investigation

The core pieces (Figure 6) deformed in the mine were cut parallel to the longitudinal axis of the core and thin sections were taken from the resulting slabs as well as samples for SEM examination.

A detailed description of the methods used for the microstructural investigations using state-of-the-art microanalysis can be found in the ZW1.2 final report, to which we refer here.

To determine grain size (Figure 7), we developed a semi-automatic segmentation tool that allowed rapid manual segmentation of grains from reflected light microphotographs of the etched thin section surfaces (the pixel resolution was $3.45 \mu\text{m}$). Grain size was calculated using the circular equivalent diameter d . Most grain boundaries and grains contained second phases such as fluid inclusions and small anhydrite grains or grain clusters. We used the segmentation routine to exclude second phases present at the grain boundary - and to include second phases present inside the grains in the measured area of the grain. Grains touching the edges of the images or thin sections were not measured.

We use subgrain size as a piezometer using the laboratory calibrated relationship between mean subgrain size and differential stress (Carter et al., 1993). The subgrain size is described as the circular equivalent diameter (D) of the segmented subgrains (Figure 8). The pixel resolution of the analysed micrographs was $0.69 \mu\text{m}$. For each thin section, several grains with subgrains at different locations in the thin section were selected. We followed the rule of only segmenting subgrains that were visible as closed polygons and were not associated with high angle grain boundaries. Subgrains touching the edges of the images were not measured.

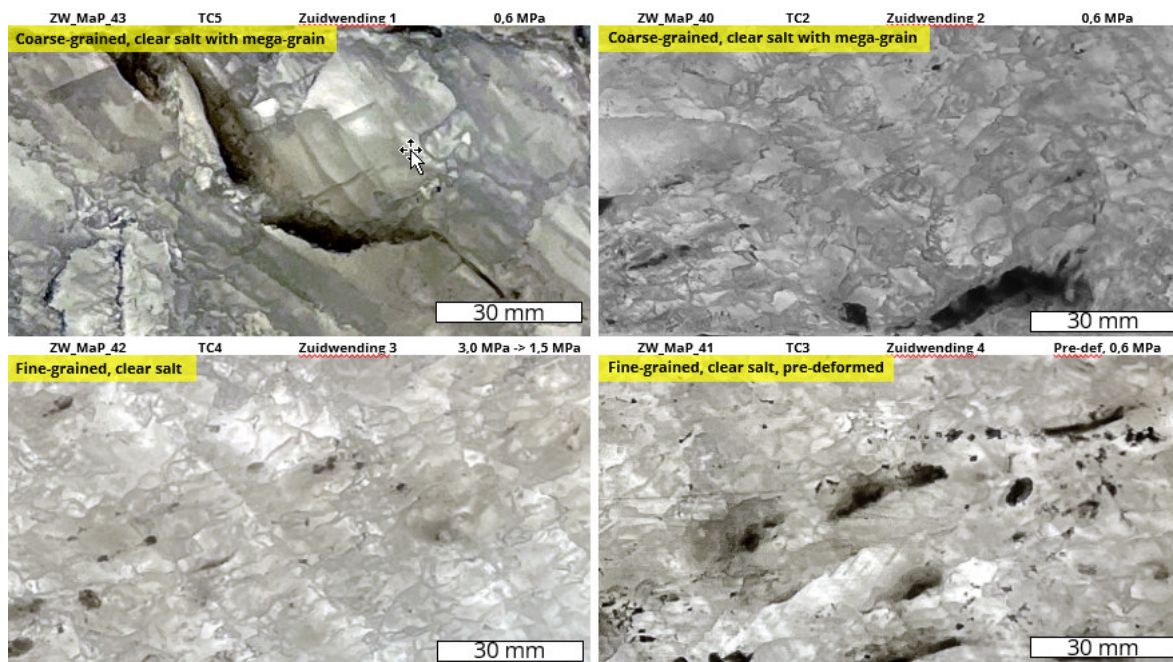


Figure 6 Transmitted light flatbed scan of the twin sample slab.

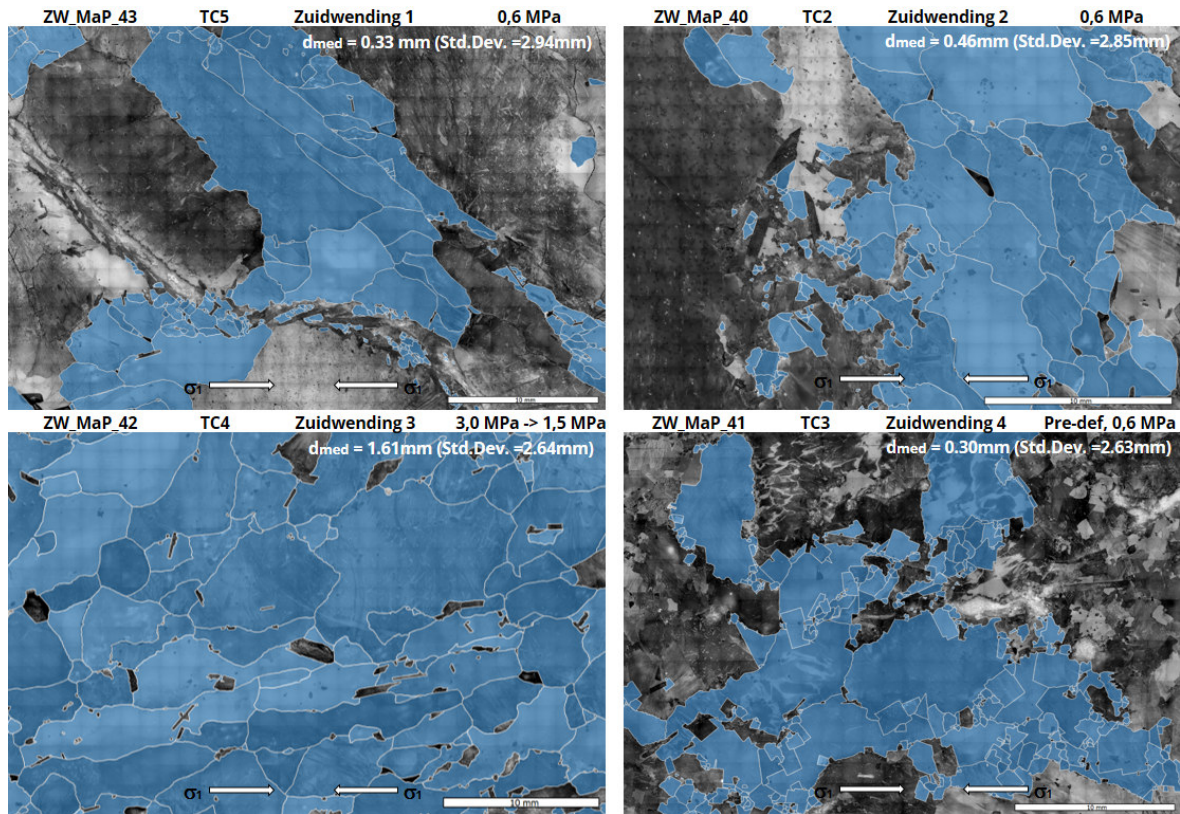


Figure 7 Reflected light micrographs with semi-transparent overlay of the grain segmentation result.

1.4 Tested samples

Four samples from Zuidwending were tested. The origins of the samples are given in Table 3.

Table 3. Origin of the tested cores.

Sample Name	Core piece ID	Depth (m)
Zuidwending_1	80_TC5	1505.9
Zuidwending_2	30_B_TC2	1401.18
Zuidwending_3	70_B_TC4	1401.58
Zuidwending_4	50_C_TC3	1401.38

Dimensions of the samples are given in Table 4. Sample Zuidwending 4 was pre-deformed at IfG laboratory.

Table 4. Properties of the tested samples.

Sample Name	Height (mm)	Diameter (mm)	Weight (g)	Density	Pre-deformed
Zuidwending_1	140.05	70.28	1173.2	2.159	no
Zuidwending_2	140.11	70.33	1191.9	2.190	no
Zuidwending_3	140.22	70.25	1185.1	2.181	no
Zuidwending_4	113.01	70.14			yes

Dimensions of the sample before and after the creep tests performed at IfG laboratory are given in Table 5.

Table 5. Size reduction of the pre-deformed sample.

Sample Name	Initial Size	Final Size	
	Height (mm)	Height (mm)	Reduction (%)
Zuidwending_4	140.17	113.01	19.4

1.5 Loads

Loads applied on each sample are given in Table 6. For the last sample that was pre-deformed at IfG laboratory, load is calculated either considering the initial height before the preliminary creep tests or considering the final sample height after this initial creep test.

Table 6. Loads on the samples.

Sample Name	Weight (kg)	Load (MPa)	
		Calculated on initial size	Calculated on final size
Zuidwending_1	233	0.61	NA
Zuidwending_2	233	0.61	
Zuidwending_4	233.3	0.51	0.61

The evolution of the load applied to samples 3 is shown in Figure 8.

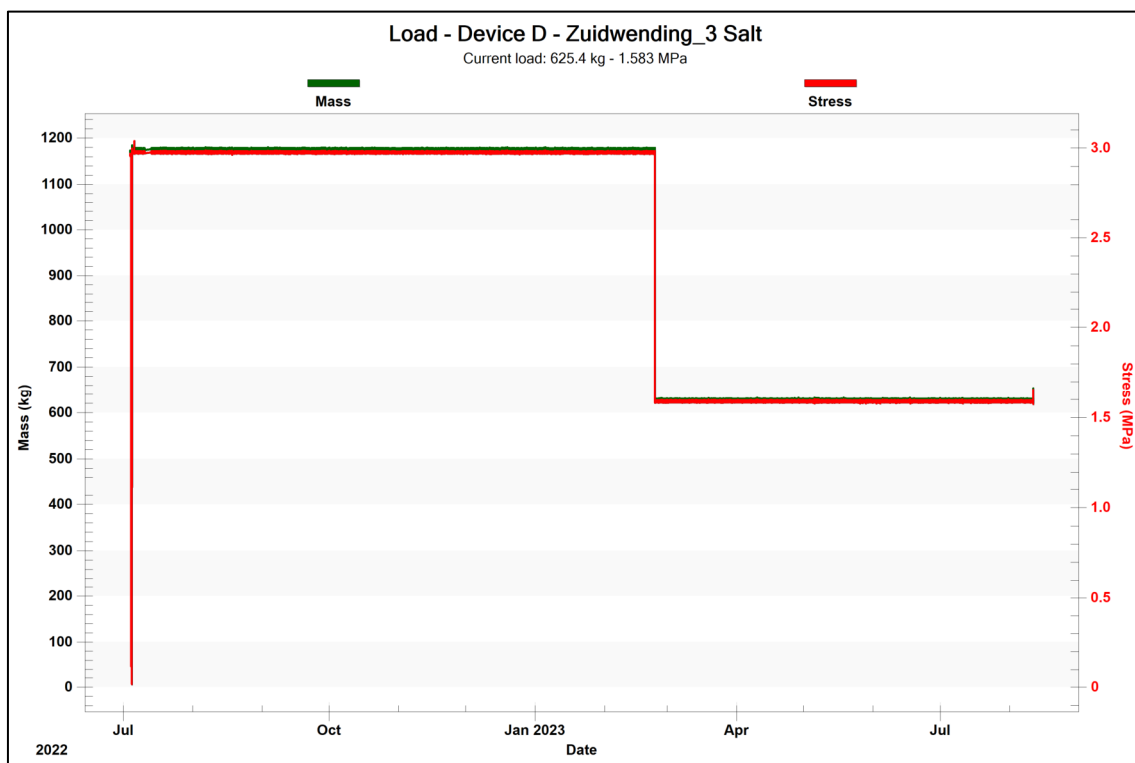


Figure 8. Evolution of load on sample #3.

2 TEST RESULTS

2.1 Data points

The testing period started on 6 July 2022 and was ended 30 August 2023. The total duration was 420 days or 14 months. Measurements were taken every minute, and a total of 604,102 measurements data points were recorded.

2.2 Measured temperature

Figure 9 shows the temperature in the gallery measured during the 9-month long testing period. The average temperature is 8 °C. The small variations around this temperature are probably due to the remote activity of the mine.

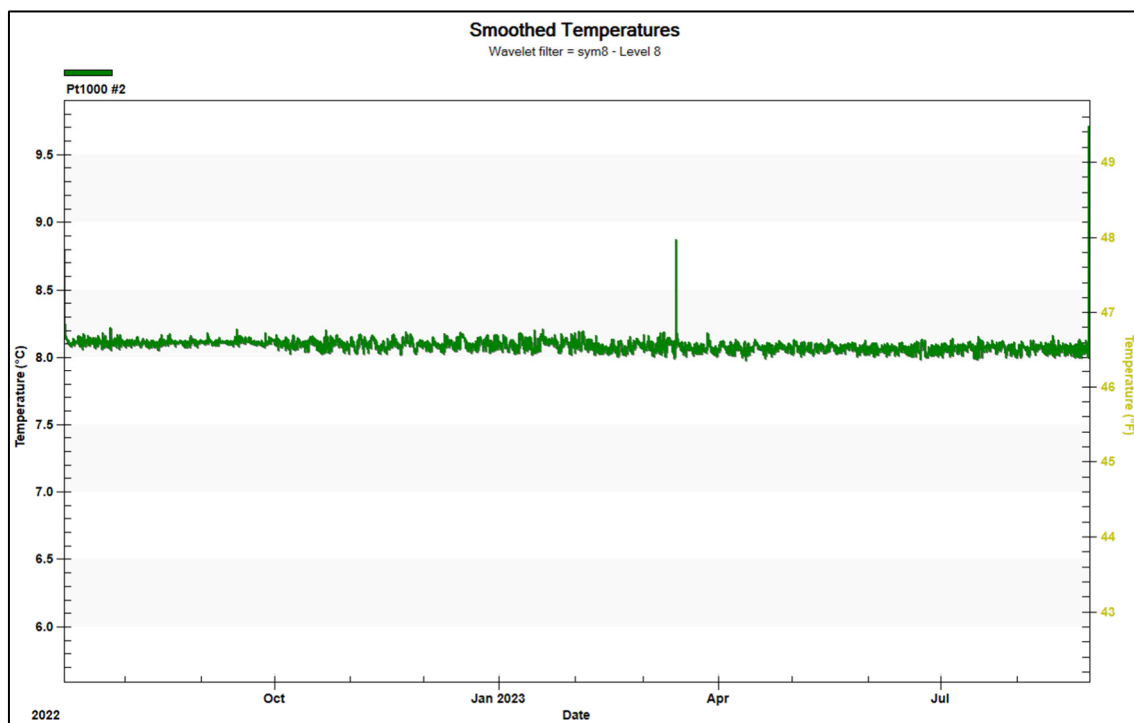


Figure 9. Temperature in the gallery measured during the testing period.

2.3 Measured humidity

Figure 10 shows the relative humidity in the gallery measured during the testing period. The average relative humidity is 67.5 %RH. A transitional period of one month can be observed at the beginning.

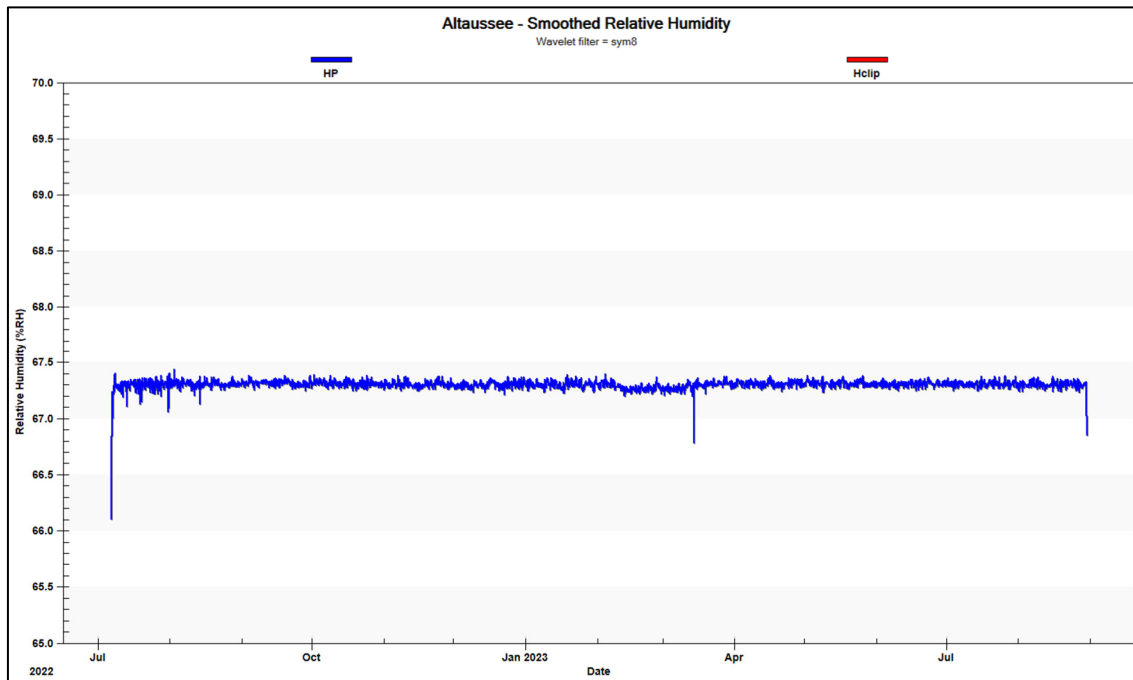


Figure 10. Relative humidity in the gallery measured during the testing period.

2.4 Measured displacements

Figure 11 to Figure 14 show the raw displacement measurements.

2.5 Calculated strains

Figure 15 to Figure 18 show the calculated strains.

2.6 Calculated strain rates

Figure 19 to Figure 22 show the calculated strain rates which are plotted both in linear and log scales. Strain rates are calculated using a weighted moving-average method. A sliding window of one week is considered. The weighting is performed using binomial coefficients. Table 7 provides the main results of the creep tests.

Table 7. Main results of the creep tests.

Sample Name	Load (MPa)	Total strain ($\times 10^{-6}$)	Final strain rate ($\times 10^{-12}$ /s)
Zuidwending_1	0.61	561	2.63
Zuidwending_2	0.61	525	2.51
Zuidwending_3	2.98 - 1.59	1060 – 37.5	8.00- 1.55
Zuidwending_4	0.61	1128	5.00

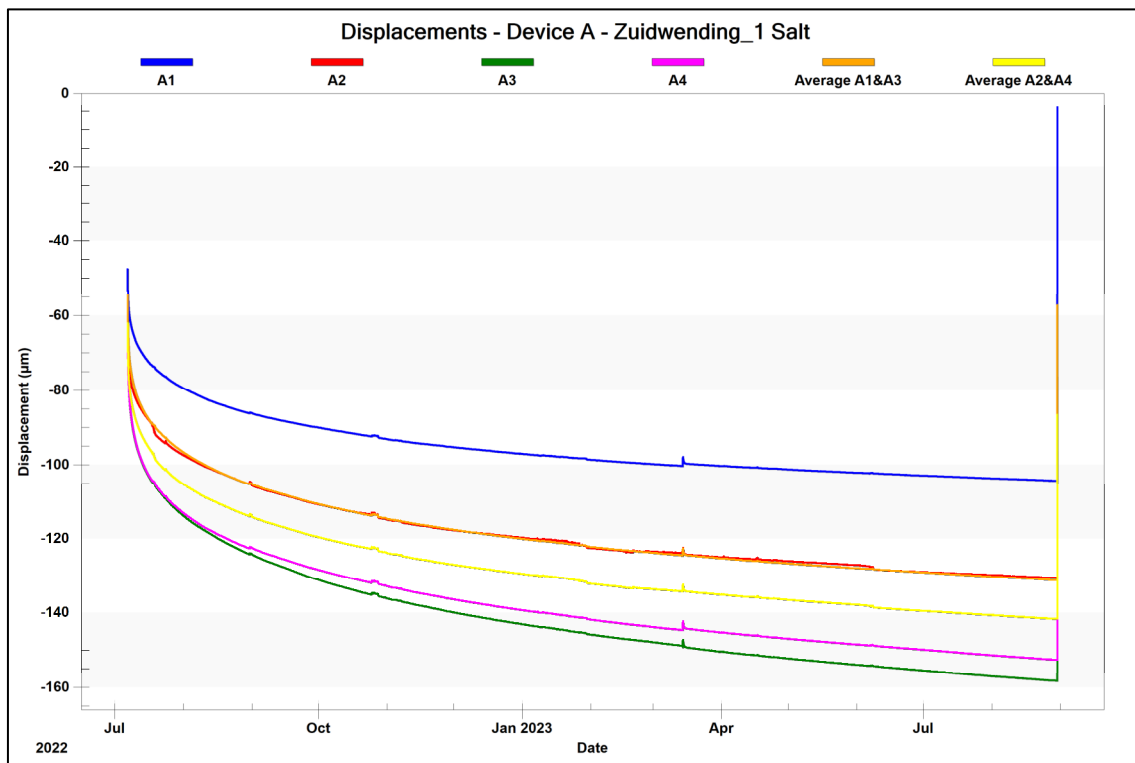


Figure 11. Sample #1 - Measured displacements.

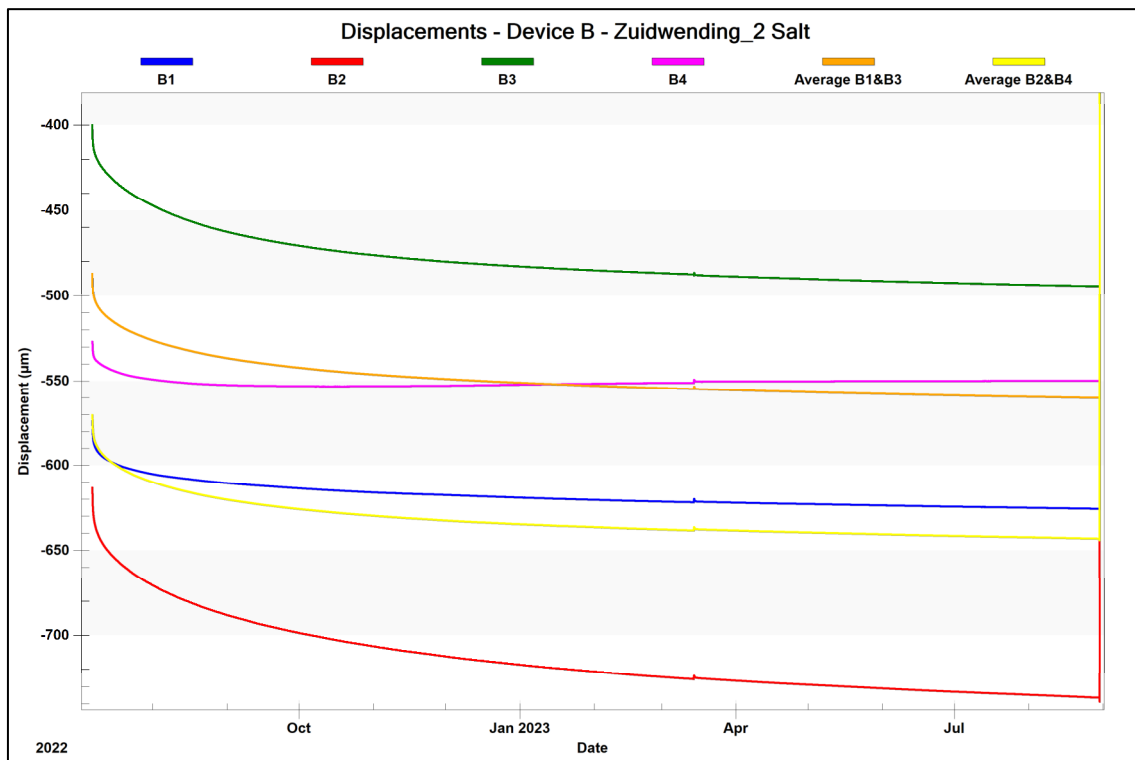


Figure 12. Sample #2 - Measured displacements.

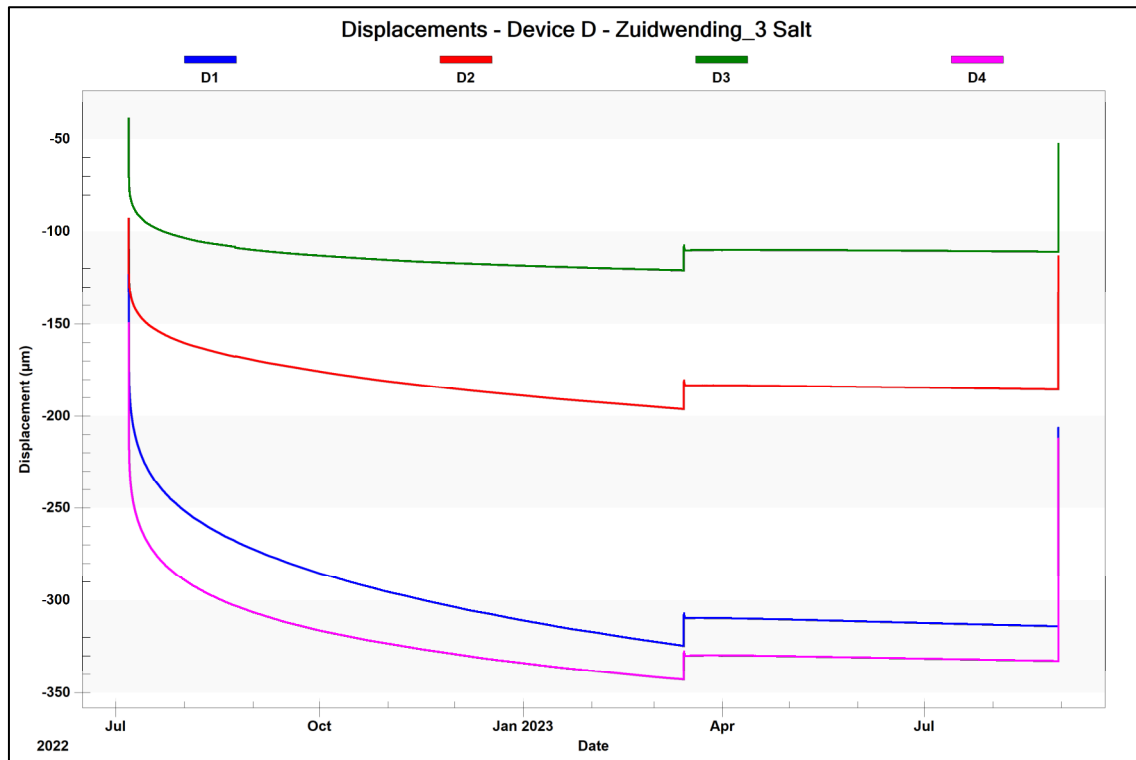


Figure 13. Sample #3 - Measured displacements.

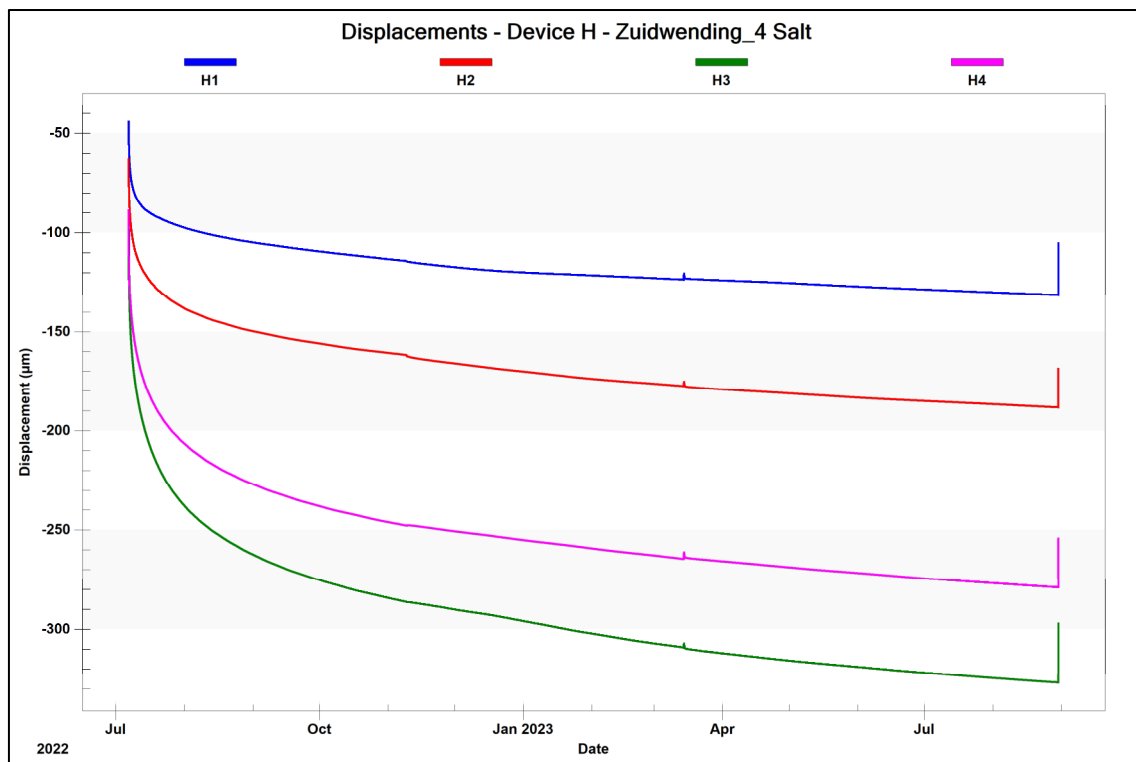


Figure 14. Sample #4 - Measured displacements.

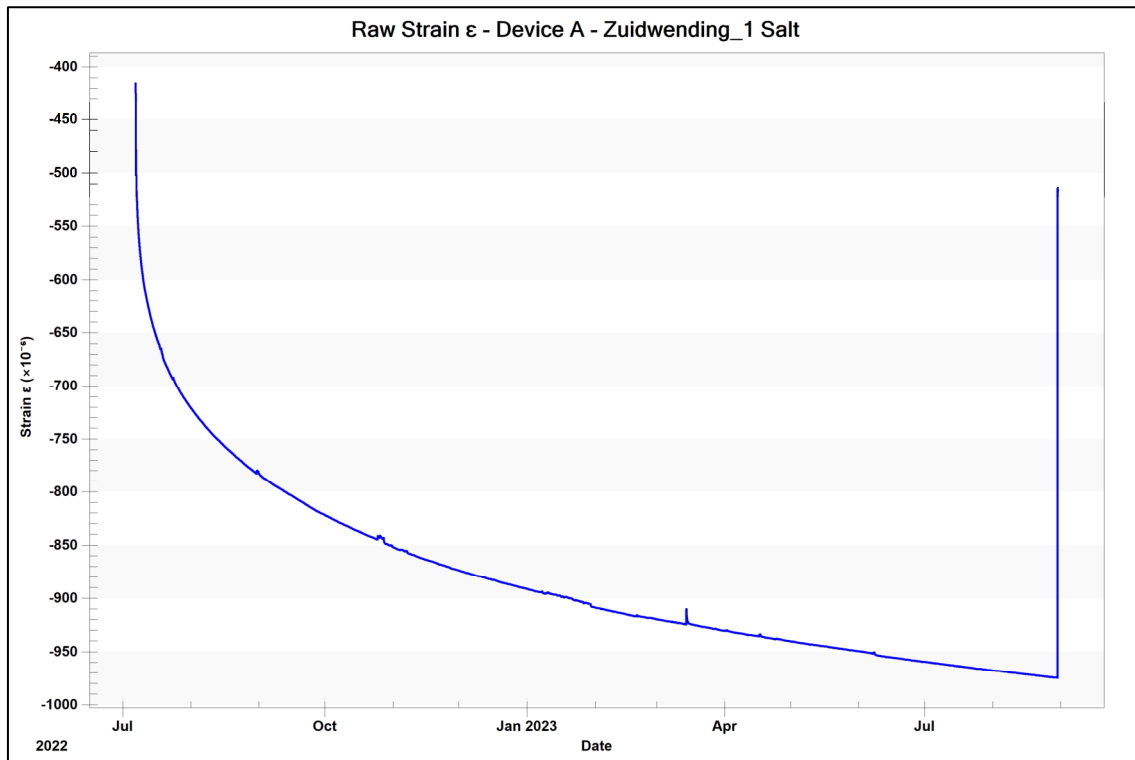


Figure 15. Sample #1 – Evolution of the strain.

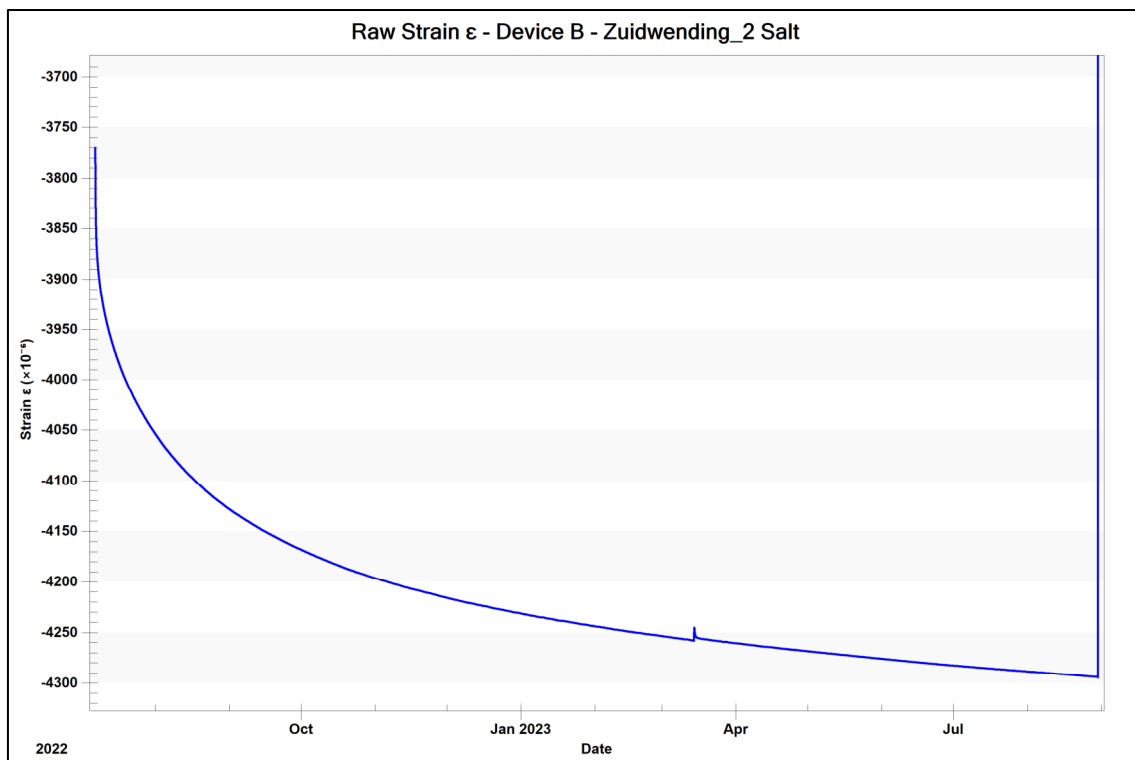


Figure 16. Sample #2 – Evolution of the strain.

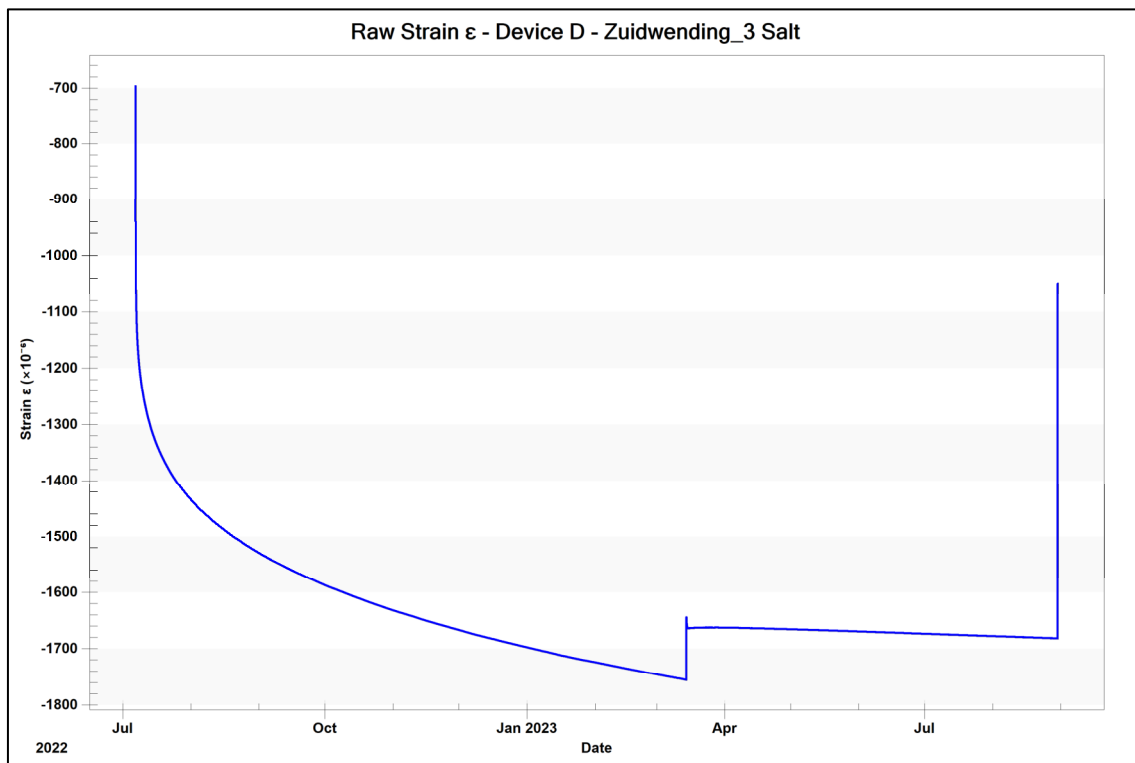


Figure 17. Sample #3 – Evolution of the strain.

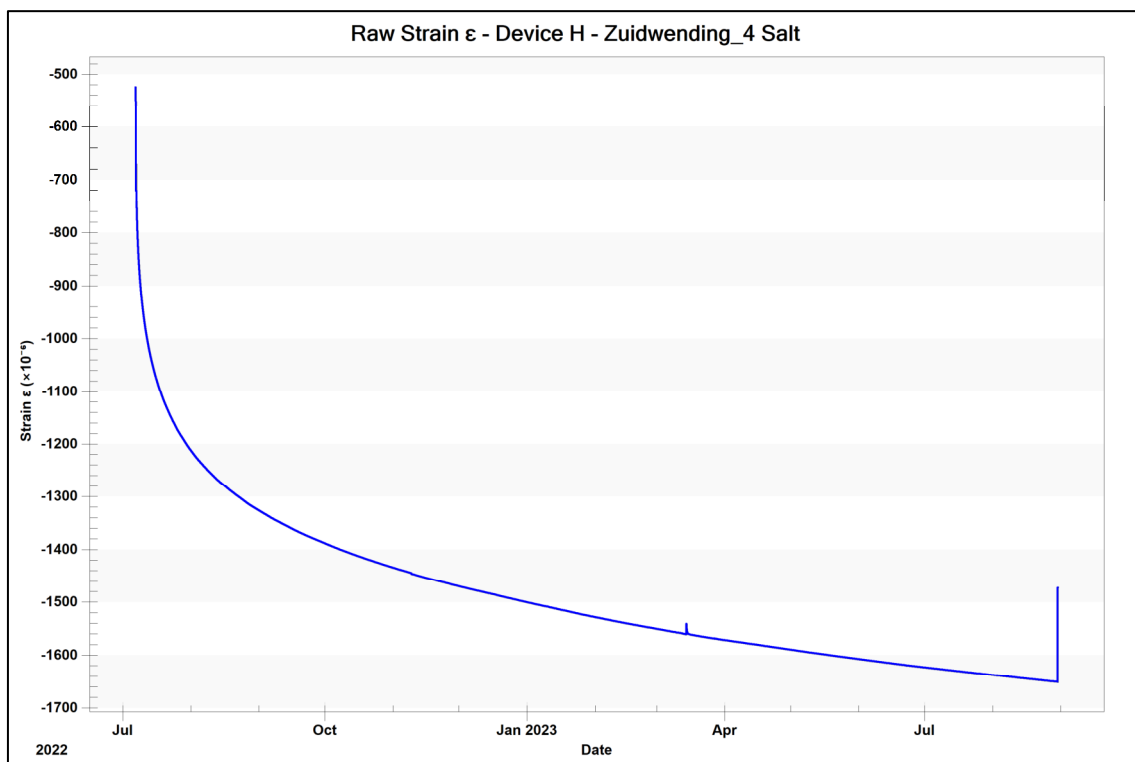
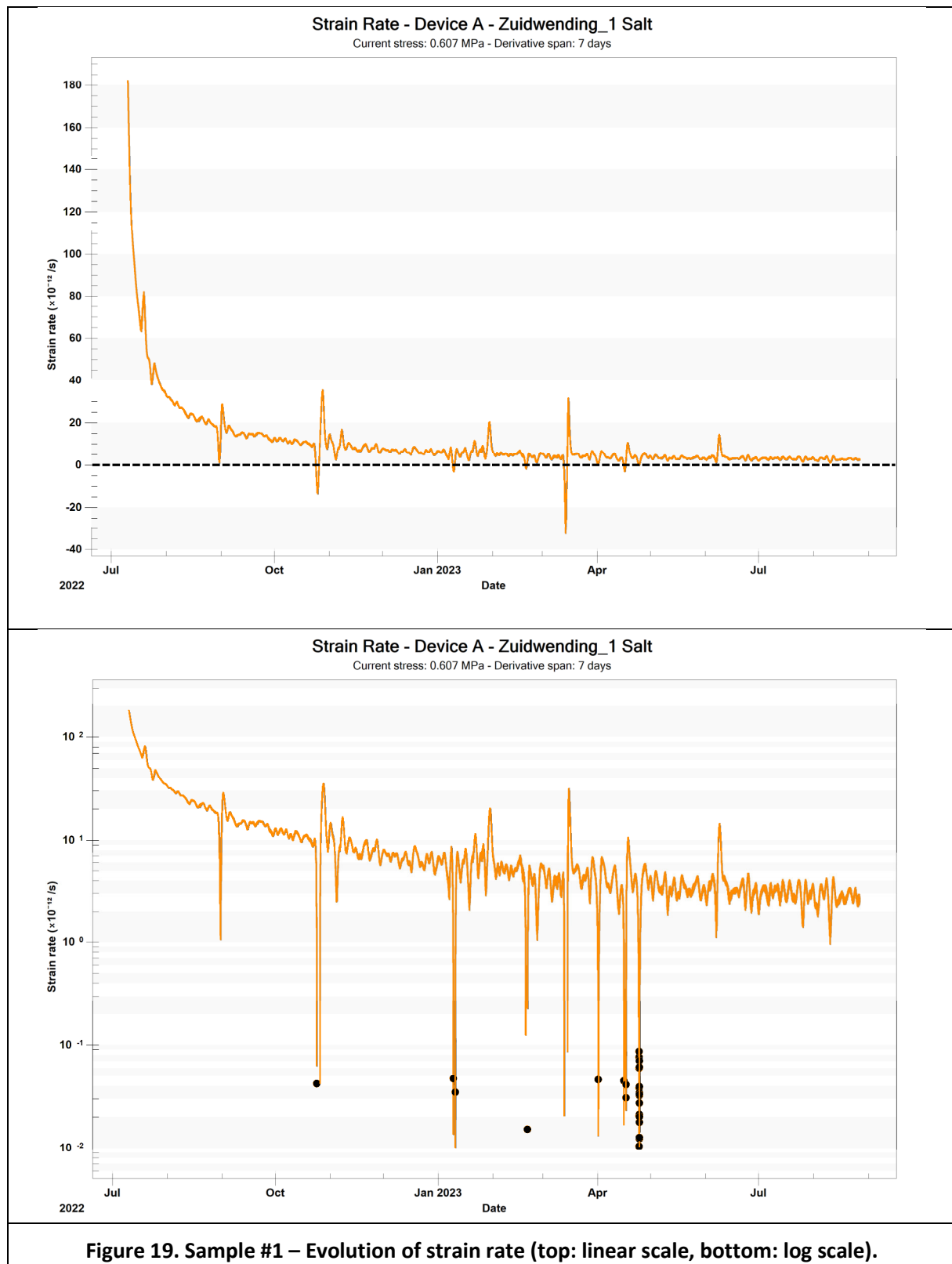
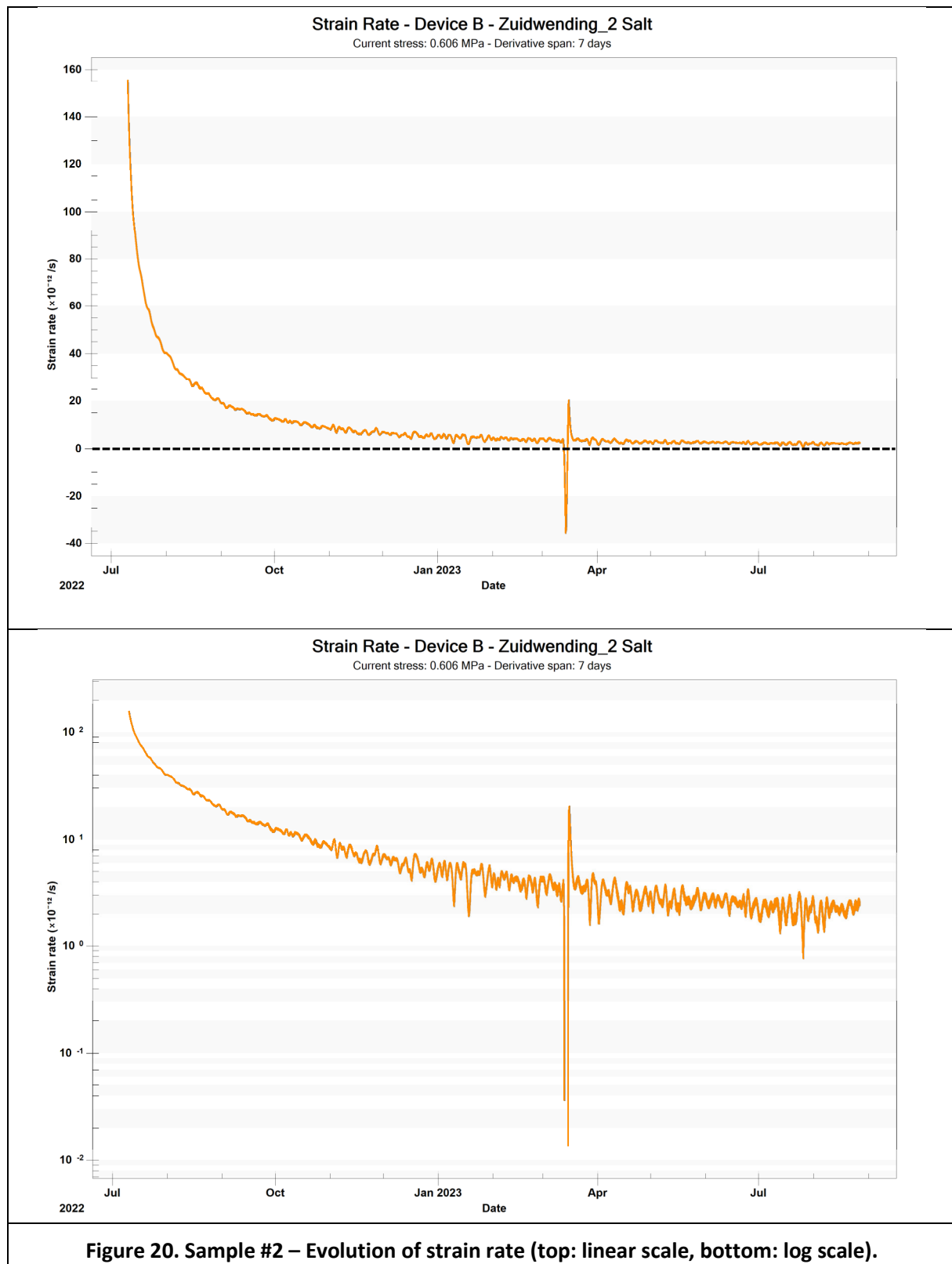
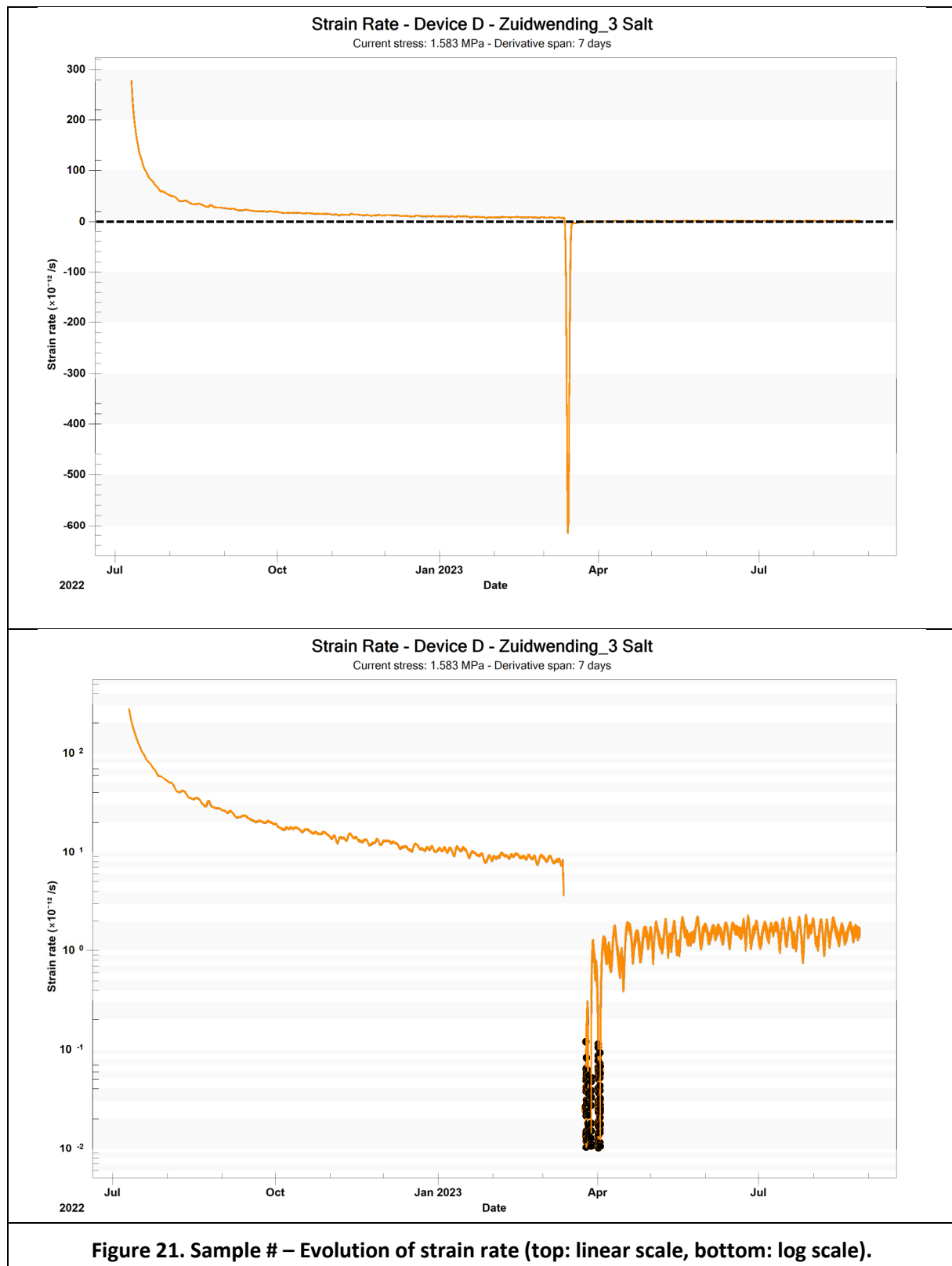
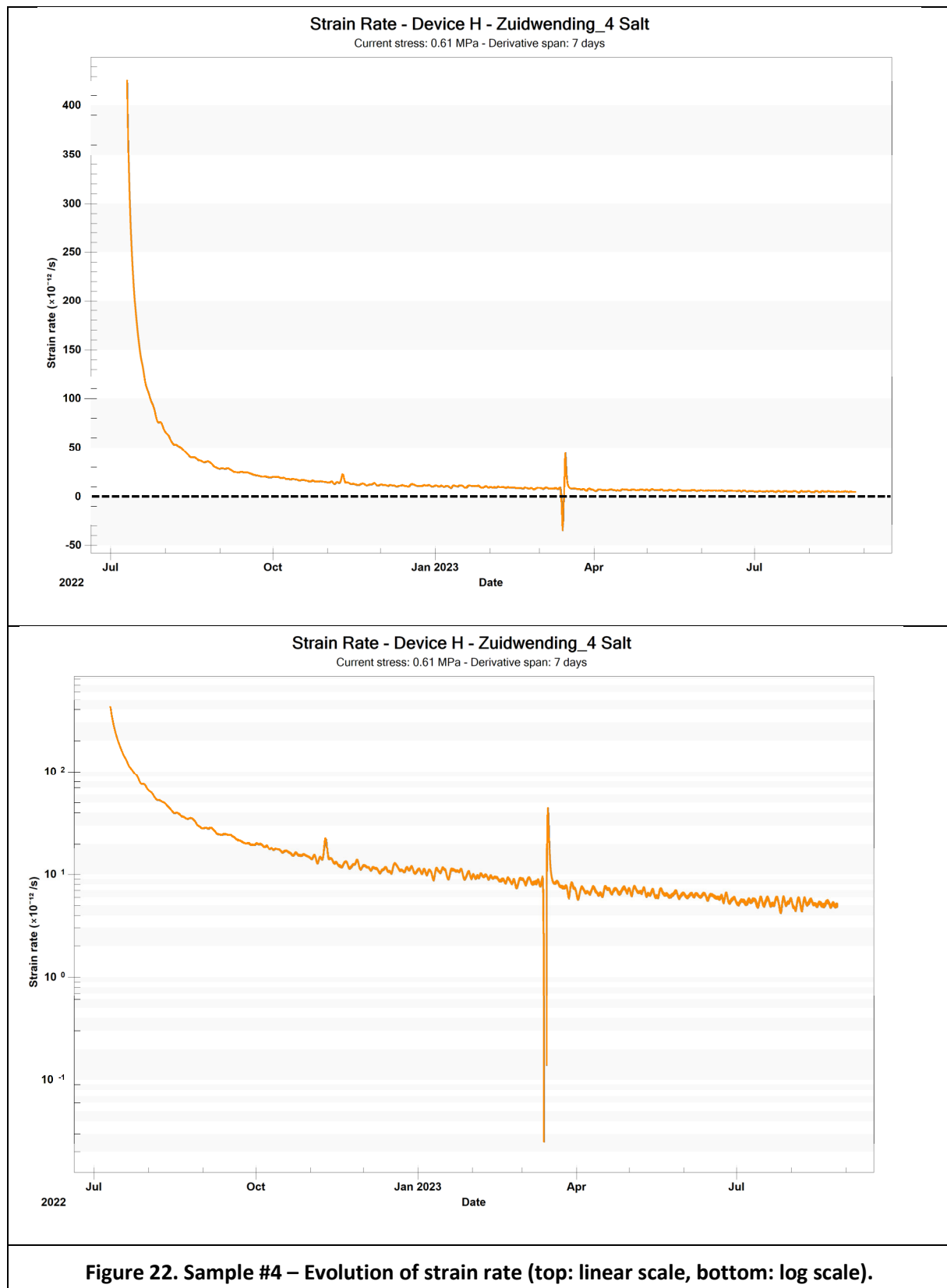


Figure 18. Sample #4 – Evolution of the strain.









2.7 Comparison of the microstructure of tectonically deformed and mine deformed samples

This work aims providing a comparison of the microstructure of the Zuidwending salt core pieces, selected for the small deviatoric-stress creep tests performed in the Altaussee Mine before and after deformation.

2.7.1 *Microstructural processes*

The macroscopic and microscopic examinations of the initial samples (refer to the ZW1.1 and ZW1.2 reports for further information) - referred to as twin samples in this report - clearly reveal the presence of three distinct structural components: (i) halite mega-grains, measuring several decimetres in length and up to 2 centimetres in thickness, with abundant fluid and second-phase inclusions enclosed within the grains, (ii) fine-grained matrix halite, and (iii) thin layers of fine-grained anhydrite, folded and boudinaged, with a thickness of a few millimetres. Dispersed anhydrite dust, visible as small idiomorphic grains or grain clusters is commonly found along matrix halite grain boundaries and enclosed within the matrix halite grains. The alignment of the mega-grains is predominantly parallel to the bedding, while the matrix halite constitutes a varying proportion of the rock. Overall, tight grain boundaries were observed, rendering the rock non-permeable, with equilibrated micropore arrays of fluid inclusions within the halite fraction. Some grain boundaries exhibited dilation due to core damage. Boudinaging of the mega-grains was evident, with the presence of subgrains indicating internal deformation via dislocation creep. The matrix halite experienced deformation through a combination of dislocation and pressure solution creep, as well as dynamic recrystallization.

The four samples (Figure 6 and Figure 23), selected for the creep-test in the mine, represent: (i) a facies composed of coarse-grained matrix halite with mega-grains (Zuidwending 1 and Zuidwending 2, litho-class F, compare with ZW1.1 report), and (ii) fine-grained matrix halite (Zuidwending 3 and Zuidwending 4, litho-class A, compare with ZW1.1 report). Prior to the creep-test, all samples were hydrostatically loaded with 20 MPa at 90°C for approximately 80 hours. Sample Zuidwending 4 was subjected to a 20% axial strain at 90°C. A small amount (~0.6 ml) of saturated NaCl-brine was added to the jacket prior to installation into the creep apparatus in the mine.

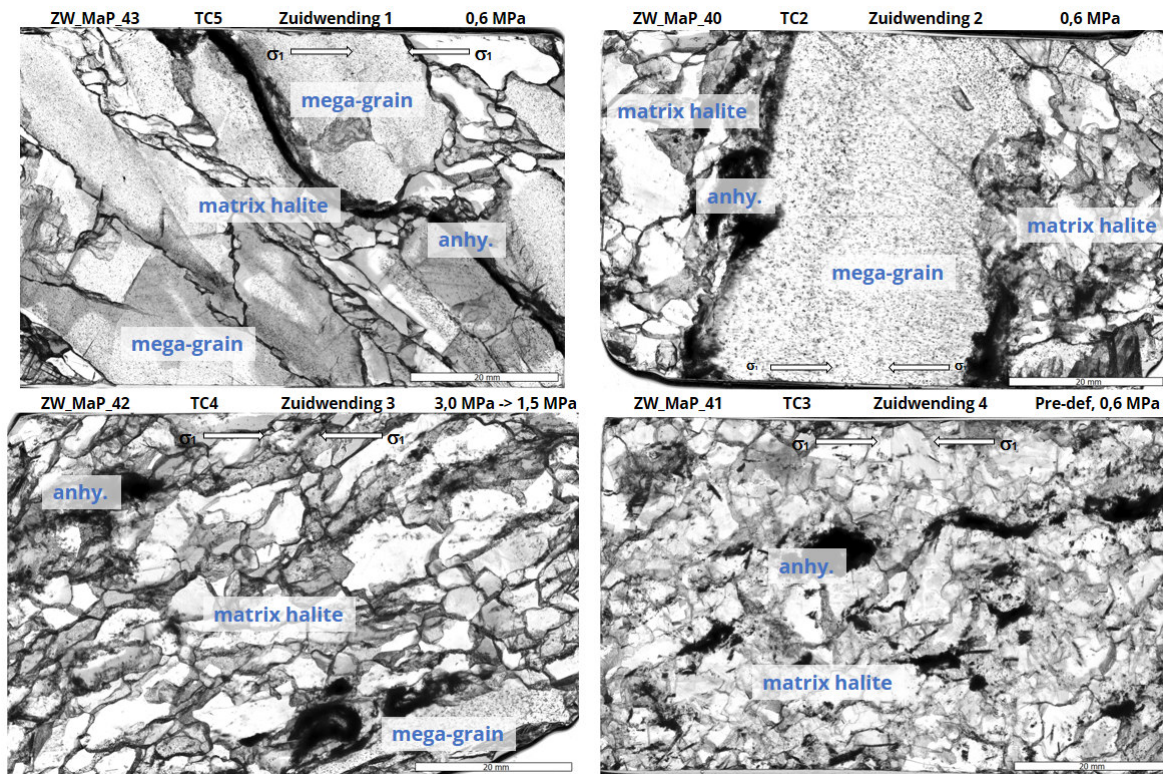
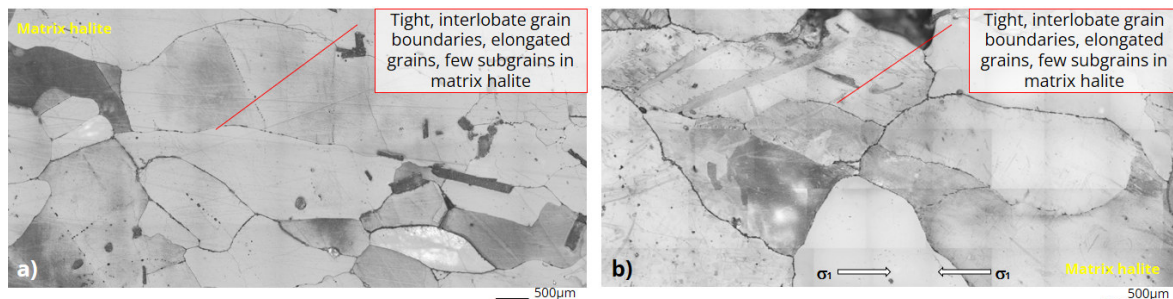


Figure 23 Thin sections prepared in the centre of the creep-tested core-pieces imaged in transmitted light.

Samples creep-tested in the mine show macroscopically the same structural elements and overall texture as the initial samples, with the exception of the pre-deformed Zuidwending 4 sample, which is partly recrystallized. This sample is believed to represent the texture after the pre-test, with modifications to grain shape occurring during the mine test. Matrix grains in samples Zuidwending 1-3 are elongated, with interlobate grain boundaries comparable to the initial samples, and the mega-grains also do not show alteration in overall shape or internal structure (see Figure 24a, b, and Figure 25). The texture of the pre-deformed sample has changed due to recrystallization processes: axial strain of 20% at 90°C allowed grain boundary migration recrystallization during the pre-test, which is recorded in the partly recrystallized texture by small strain-free grains that preferentially nucleated at existing grain boundaries and grew at the expense of neighbouring grains with higher stored energy (Figure 24c, d). The resulting textural change is a refined grain size around preserved initial grains.



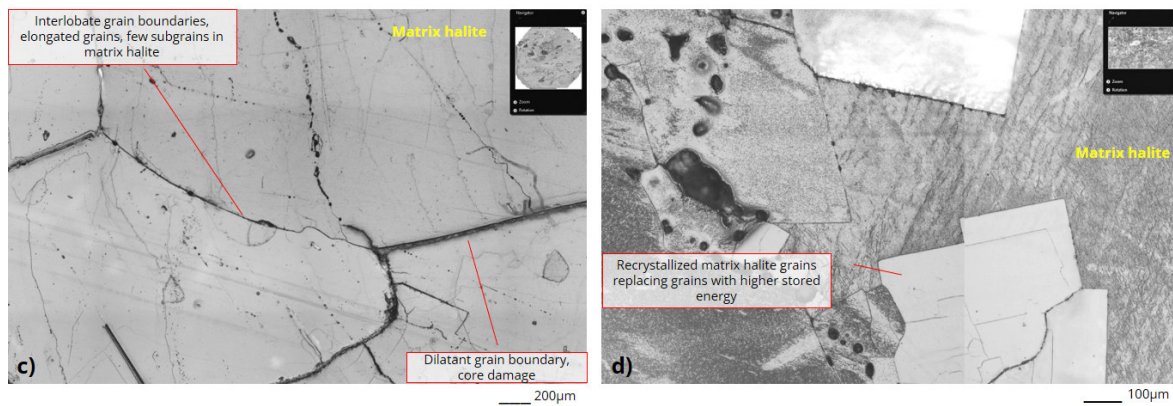


Figure 24 Reflected light micrographs are utilized to juxtapose the twin samples with the creep-deformed samples, demonstrating changes in the overall texture. The specimens presented include a) twin ZW_MaP_04, b) Zuidwending 1, c) ZW_MaP_05, and d) Zuidwending 4 (pre-deformed). Further examples for all four creep-tested samples are available in the Appendix. Please refer to the text for detailed explanations.

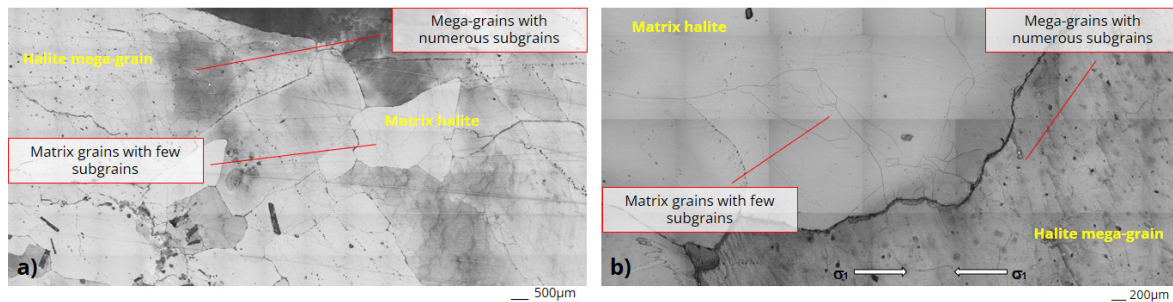


Figure 25 Reflected light micrographs are utilized to juxtapose the twin samples ZW_MaP_04 with the creep-deformed sample Zuidwending, pointing to minor differences regarding the appearance of subgrains within matrix halite and halite mega-grains. Further examples for all four creep-tested samples are available in the Appendix.

The grain boundary structure, however, has visibly changed when comparing the initial twins with the creep-deformed samples. While grain boundaries in the initial samples are commonly smooth, creep-tested samples often exhibit serrated grain boundaries (refer to Figure 26). Preferably, grain boundaries that contain large fluid or second-phase inclusions are affected by pinning processes, sometimes resulting in grain boundary bulges (see Figure 26d). Serrated grain boundaries seem to form preferably perpendicular to the orientation of the maximum principal stress of the creep test, which could indicate dissolution as a result of increased contact stress. Furthermore, it was observed that most grain boundaries in the creep-tested samples showed efflorescence (refer to Figure 26b and Figure 27b), with NaCl precipitates on the sample surfaces along the grain boundary visible after thin section preparation. This is thought to represent in-situ grain boundary fluids that reach the sample surface due to capillary forces resulting from sample preparation (Schenk and Urai, 2004; Desbois et al., 2012). Efflorescence is rarely observed in thin sections of the initial samples (see Figure 27a for an example).

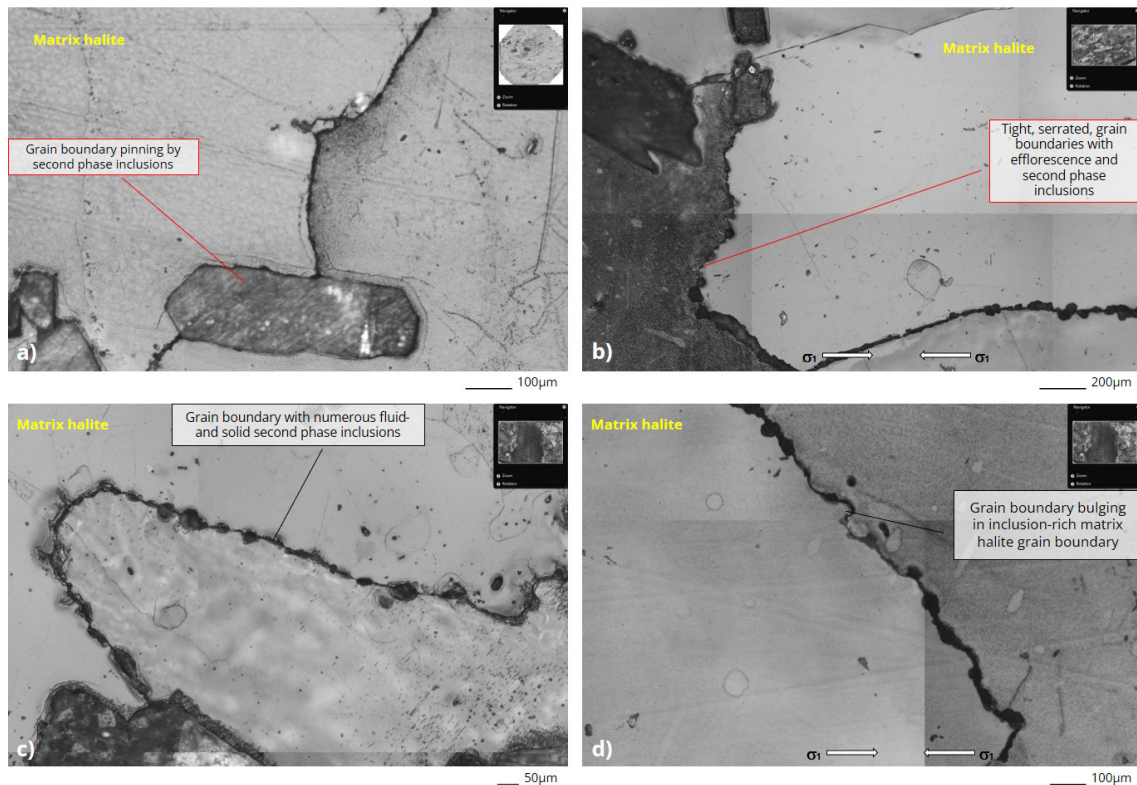


Figure 26 Reflected light micrographs are employed to juxtapose the twin samples with the creep-deformed samples, revealing the grain boundary structure: a) and c) ZW_MaP_05, b) Zuidwending 3, d) Zuidwending 2. Further examples for all four creep-tested samples are available in the Appendix. Please refer to the text for detailed explanations.

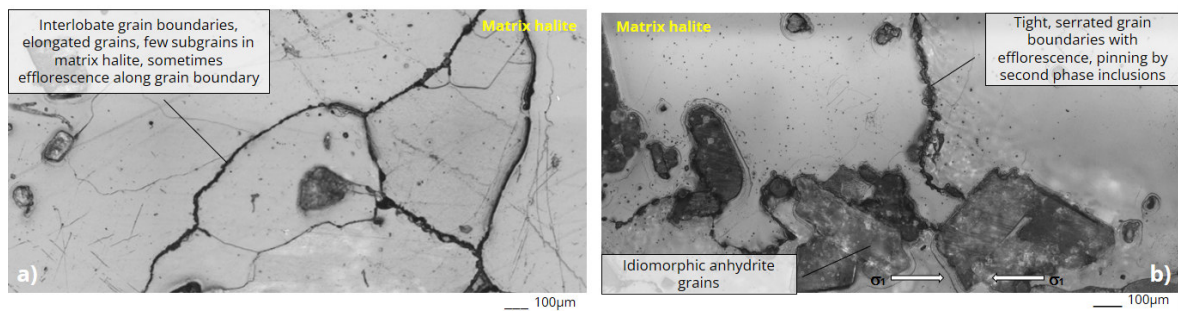


Figure 27 Reflected light micrographs are employed to juxtapose the twin samples with the creep-deformed samples, revealing the grain boundary structure: a) ZW_MaP_05 and b) Zuidwending 2. Further examples for all four creep-tested samples are available in the Appendix. Please refer to the text for detailed explanations.

Transmitted light microscopy was employed to study the nature and distribution of grain boundary fluids in thin section samples. Comparing the samples creep-deformed in the mine, which were not pre-deformed, with their initial state (refer to Figure 28), it becomes evident that the arrays of fluid inclusions in the creep-tested samples are larger and patchier compared to the well-equilibrated fluid inclusions of the initial samples. However, grain boundaries of the recrystallized grains within the pre-deformed sample Zuidwending 4 appear to be mostly depleted in grain boundary fluids, and are thus often invisible in transmitted light (see Figure 28c). Nevertheless, in some instances, the recrystallized grains contain a large amount of fluid inclusions in comparable proportions to the non-pre-deformed samples.

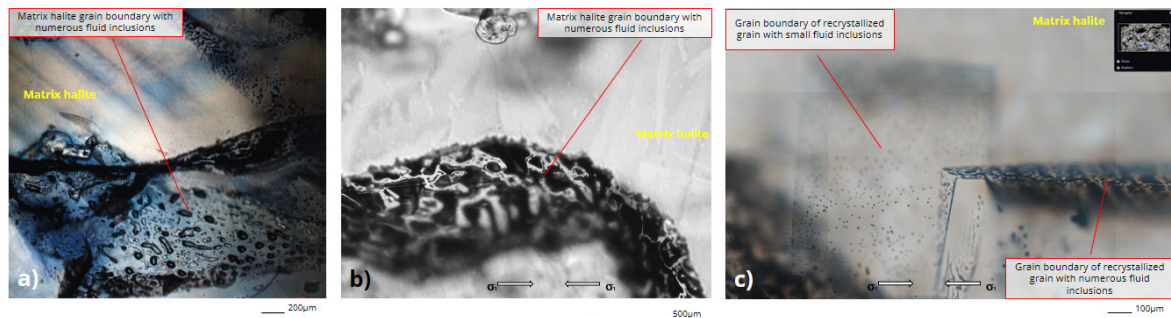


Figure 28 Transmitted light micrographs are employed to juxtapose the twin samples with the creep-deformed samples, showing the distribution of grain boundary fluid inclusion arrays a) in the initial sample ZW_MaP_05, b) the creep-tested sample Zuidwending 3, and c) the pre-deformed creep-tested sample Zuidwending 4. Further examples for all four creep-tested samples are available in the Appendix. Please refer to the text for detailed explanations.

High-resolution SEM was employed on halite polycrystals, broken along grain boundaries, to investigate grain surface morphology, imprints of fluid inclusion arrays into the grain surfaces, as well as the interfaces of adjacent grains. Compared to the initial samples, much fewer imprints of regular fluid inclusions are observed on the grain surfaces of the creep-tested samples. The observed imprints point to more channel-like or patchy grain boundary fluid inclusions compared to the initial samples, where imprints often indicated the presence of well-equilibrated, regular arrays of fluid inclusions along the grain boundaries, also visible in the transmitted light micrographs (refer to the Appendix for examples).

Along grain interfaces, efflorescence was observed, indicating the precipitation of grain boundary fluids during sample preparation. In addition to these processes, as reported by Schenk and Urai (2004) and Desbois et al. (2012), which relate the occurrence of NaCl precipitates along the grain boundaries to the release of grain boundary fluids during sample preparation, the structure of the observed precipitates at the interface of adjacent grains, as seen using SEM (see Figure 29), indicates generations of precipitates recorded in structures reminiscent of syntaxial vein growth. The structures formed on the grain surfaces vary from amorphous to fibrous to blocky crystal shapes, sometimes accompanied by porosity (refer to Figure 29b,c). These observed structures may indicate a process of grain boundary opening, facilitating the precipitation of NaCl depending on the concentration gradient in a dissolution-precipitation process (Urai and Spiers, 2007, and references therein). Furthermore, locations with grain overgrowths in grain triple junctions are occasionally visible (Figure 30).

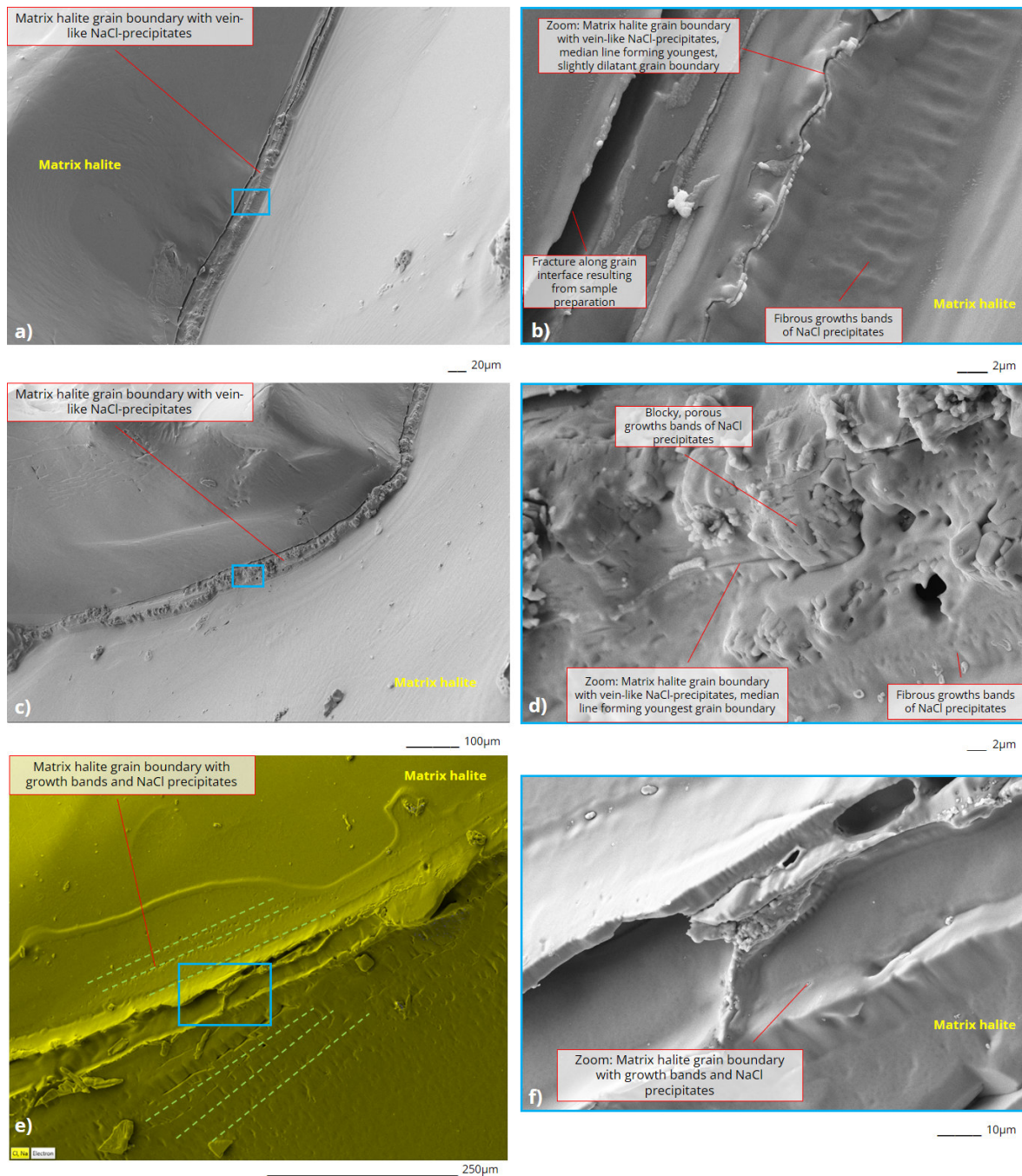


Figure 29 SEM micrographs showing grain surfaces and contacts of adjacent grains in sample Zuidwending 3: b) is detail of a), d) is detail of c), f) is detail of e) with EDS overlay and green, dashed lines indicating growths bands. Further examples for all four creep-tested samples are available in the Appendix. Please refer to the text for detailed explanations.

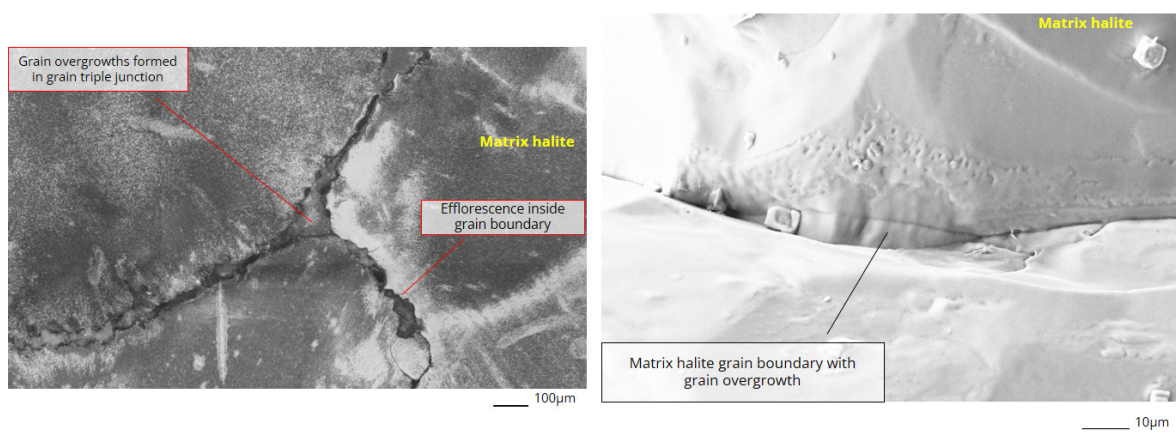


Figure 30 Reflected light thin section micrograph (left) and SEM micrograph of the grain surface, revealing grain overgrowth formed at grain triple junction and along matrix halite grain boundary.

2.7.2 Grain size distribution

The average median grain size (refer to Table 8, Figure 31) measured in sample ZW_MaP_04, which is a twin to Zuidwending 1, is 1.06 mm (Number 440, Stddev. 2.21 mm), while the average median grain size for the creep-tested sample Zuidwending 1 is 0.34 mm (Number 501, Stddev. 1.14 mm). The twin sample ZW_MaP_05 has an average median grain size of 1.05 mm (Number 507, Stddev. 1.54 mm). The related creep-tested samples Zuidwending 2, 3, and 4 (pre-deformed) have average median grain sizes of 0.46 mm (Number 501, Stddev. 1.19 mm), 0.61 mm (Number 502, Stddev. 1.78 mm), and 0.30 mm (Number 506, Stddev. 0.84 mm), respectively, pointing to grain refinement processes in all creep-tested samples. The tectonically deformed twin samples ZW_MaP_04 and ZW_MaP_05, as well as the pre-deformed sample Zuidwending 4, have a log-normal distribution of grain size, while samples Zuidwending 1-3 show a log-normal distribution of grain size with a negative skew.

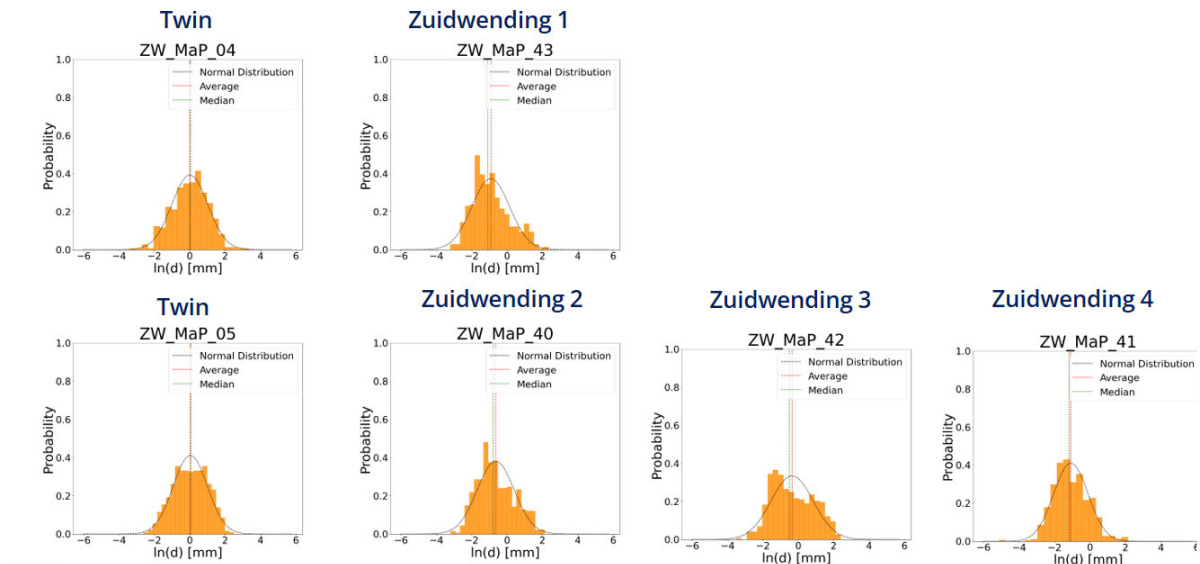


Figure 31 Grain size distribution histograms

Table 8

Mine ID	Well information	Thin section ID	Count	Min [mm]	Max [mm]	Median [mm]	Average [mm]	Std_deviation [mm]	Mode [mm]
Zuidwending 1	ZW A2A_8_7	ZW1_2_MaP_43	501	0.04	10.43	0.34	0.76	1.14	0.19
Twin ZW1.2	ZW A2A_8_7	ZW_MaP_04	440	0.03	27.19	1.06	1.64	2.12	0.31
Zuidwending 2	ZW_A7B_1_13	ZW2_3_MaP_40	501	0.04	8.37	0.46	0.94	1.19	0.84
Zuidwending 3	ZW_A7B_1_13	ZW1_2_MaP_42	502	0.03	10.86	0.61	1.41	1.78	2.01
Zuidwending 4	ZW_A7B_1_13	ZW1_2_MaP_41	506	0.01	8.44	0.3	0.57	0.84	1.8
Twin ZW1.2	ZW_A7B_1_13	ZW_MaP_05	507	0.07	12.18	1.05	1.6	1.54	0.38

2.7.3 Subgrain size distribution and inferred maximum differential stress

In general, only few subgrains are observable within the matrix grains as well as within the megagrain. Subgrain size is sensitive to stress, strain, and temperature and may have changed during the hydrostatic pre-preparation at 90°C, or during the mine test.

The average subgrain size (refer to Table 9, Figure 32) measured in sample ZW_MaP_04, twin to Zuidwending 1, is 130 μm (Number 457, Stddev. 98 μm), while the average subgrain size for the creep-tested sample Zuidwending 1 is 108 μm (Number 501, Stddev. 95 μm). The twin sample ZW_MaP_05 has an average subgrain size of 104 μm (Number 505, Stddev. 82 μm). The related creep-tested samples Zuidwending 2, 3, and 4 (pre-deformed) have an average subgrain size of 152 μm (Number 276, Stddev. 141 μm), 121 μm (Number 347, Stddev. 101 μm), and 72 μm (Number 402, Stddev. 60 μm), respectively.

We used the correlation equation proposed by Schleder and Urai (2005), based on data from Carter et al., 1993, to estimate the differential stress from subgrains measured in the reflected light micrographs. Considering the uncertainties in the calibrations and differences in measurement methods, the results imply that the maximum differential stress recorded in the microstructure was in the range of 1.3-2.1 MPa in the tectonically deformed samples, around 1.0-1.9 MPa in non-pre-deformed, creep-tested samples, while the subgrain size in the pre-deformed samples indicates differential stresses of up to 2.3 – 2.9 MPa. Overall, the inferred maximum differential stress has decreased for the creep-tested samples in comparison to the tectonically deformed samples. However, due to the small number of studied samples, as well as the minor visibility of subgrains in the creep-tested samples, it is difficult to draw a conclusion on the result of the subgrain size piezometry.

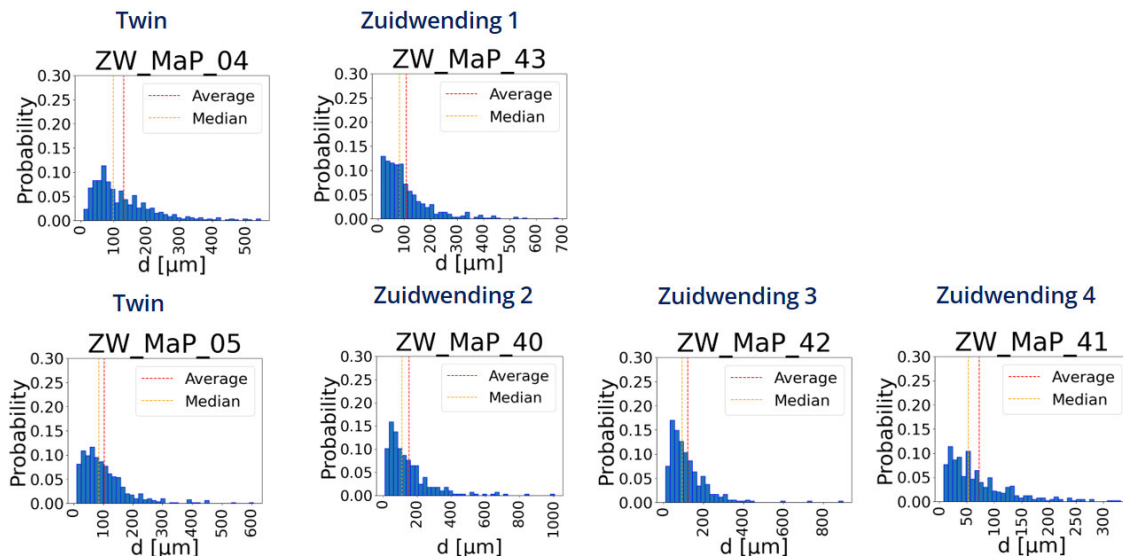


Figure 32 Subgrain size distribution histograms

Table 9

Mine ID	MaP_ID	D [μm]							Piezometer
		Number	Min [μm]	Max [μm]	Median [μm]	Average [μm]	Std_deviation [μm]	Mode [μm]	diff. Stress [MPa]
Zuidwending 1	ZW_MaP_43	501	11	683	82	108	95	14	1.81
Twin ZW1.2	ZW_MaP_04	457	9	545	100	130	98	58	1.54
Zuidwending 2	ZW_MaP_40	276	11	1005	110	152	141	92	1.35
Zuidwending 3	ZW_MaP_42	347	9	898	94	121	101	177	1.64
Zuidwending 4	ZW_MaP_41	402	7	331	53	72	60	117	2.58
Twin ZW1.2	ZW_MaP_05	505	10	606	84	104	82	65	1.88

3 CONCLUSIONS

Low load creep tests were carried out on four salt samples from the Zuidwending site. One of the samples was pre-deformed at the IfG lab in Germany. All tests went well, and the data can be considered of very good quality. The main results of the tests are given in Table 10.

Table 10. Main results of the creep tests.

Sample Name	Ref.	Pre-deformed	Median grain size (mm)	Load (MPa)	Final strain rate ($\times 10^{-12}$ /s)
Zuidwending_1	80_TC5		0.34	0.61	2.63
Zuidwending_2	30_B_TC2		0.46	0.61	2.51
Zuidwending_3	70_B_TC4		0.61	2.98 - 1.59	8.00- 1.55
Zuidwending_4	50_C_TC3	×	0.30	0.61	5.00

Figure 33 shows the results of the Zuidwending salt tests in comparison with other measurements in the literature. It should be borne in mind that the tests were carried out at a temperature of around

8 °C (Figure 9) and that therefore the creep rate would be greater at the temperature of the salt at the depth of the caverns, which can be estimated to be around 30 to 40 °C.

It is difficult to certify that the stationary strain rate has been reached at the end of each stage. For the low loads (samples 1 and 4), it is likely that this velocity has not yet been reached, but the data obtained up to this point can be used by smartTectonics for the extrapolation of a relevant parameter set.

Pressure solution creep can be described by equation (2).

$$\dot{\varepsilon}_{ps} = \frac{A_{ps}}{TD^3} \exp\left(-\frac{Q_{ps}}{RT}\right) \sigma \quad (2)$$

where A_{ps} is a prefactor, Q_{ps} is activation energy and D is average grain size.

When a $Q = 24.2$ kJ/mol is considered, the following set of parameters can be back calculated from the tests on the non-pre-deformed samples 1 and 2:

$$\begin{cases} A_{ps} = 250 - 414 \text{ K} \cdot \text{mm}^3 / \text{MPa} \cdot \text{yr} \\ D = 0.7 \text{ mm} \\ Q_{ps} / R = 2910 \text{ K} \end{cases}$$

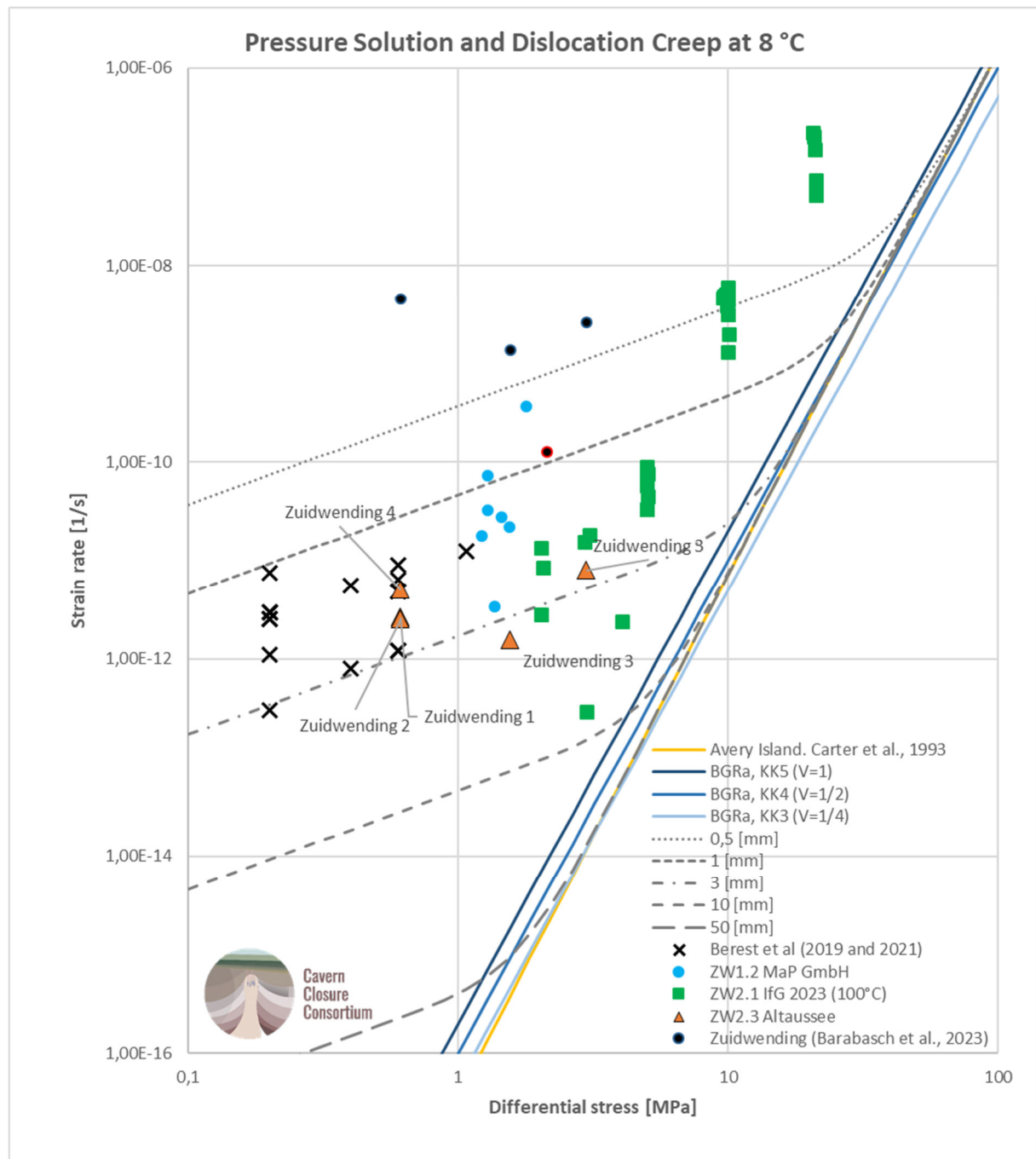


Figure 33. Comparison with other salts in the literature (Zuidwending=orange triangles).

REFERENCES

- Bérest, P., Blum, P.A., Charpentier, J.P., Gharbi, H., Valès, F. (2004). Fluage du sel gemme sous très faibles charges. *CR GEOSCI*, 336, 1337-1344.
- Bérest, P., Blum, P.A., Charpentier, J.P., Gharbi, H. and Valès, F. (2005). Very slow creep tests on rock samples, *International Journal of Rock Mechanics and Mining Sciences*, 42(4), pp. 569–576. Available at: <https://doi.org/10.1016/j.ijrmms.2005.02.003>.
- Bérest, P., Brouard, B., Karimi-Jafari, M. (2009). The effect of small deviatoric stresses on cavern creep behavior. 9th Int. Symp. on Salt, 574–589.
- Bérest, P., Béraud, J.-F., Brouard, B., Blum, P.-A., Charpentier, J.-P., de Greef, V., Gharbi, H., Valès, F. (2010). Very slow creep tests on rock samples. *EPJ Web of Conferences*, 6, 22002. <https://doi.org/10.1051/epjconf/20100622002>
- Bérest, P., Béraud, J.-F., Bourcier, M., Dimanov, A., Gharbi, H., Brouard, B., DeVries, K., Tribout, D. (2012). Very slow creep tests on rock samples. *Proceedings of 7th Conference on the Mechanical Behavior of Salt*. 7th Conference on the Mechanical Behavior of Salt, Paris, France.
- Bérest, P., Béraud, J. F., Gharbi, H., Brouard, B., DeVries, K. (2014). A Very Slow Creep Test on an Avery Island Salt Sample. *48th US Rock Mechanics / Geomechanics Symposium*. <https://onepetro.org/ARMAUSRMS/proceedings-abstract/ARMA14/All-ARMA14/ARMA-2014-7052/122841>
- Bérest, P., Gharbi, H., Brouard, B., Hofer, G., Stimmisher, S., Bruckner, D., DeVries, K., Hévin, G., Spiers, C.J., Urai, J. (2017). SMRI Research Report RR2017-1: Very Slow Creep Tests as a Basis for Cavern Stability Analysis.
- Bérest, P., Manivannan, S. (2019). Behavior of a salt cavern when creep law is modified to account for low deviatoric stresses. *Proceedings ARMA 2019*, New York, Paper 19-477.
- Bérest, P., Gharbi, H., Brouard, B., Brückner, D., DeVries, K., Hévin, G., Hofer, G., Spiers, C., Urai, J. (2019). Very Slow Creep Tests on Salt Samples. *Rock Mechanics and Rock Engineering*, 52(9), pp. 2917–2934. Available at: <https://doi.org/10.1007/s00603-019-01778-9>.
- Bérest, P., Gharbi, H., Brouard, B., Hofer, G., Stimmisher, S., Bruckner, D., DeVries, K., Hévin, G., Spiers C.J., Urai J. (2020). *Very Slow Creep Tests as a Basis for Cavern Stability Analysis – Phase 2*. SMRI Research Report RR2020-1. Solution Mining Research Institute.
- Bérest, P., Gharbi, H., Gordeliy E., Jehanno, D., Peach, C., Brouard, B., Blanco-Martin, L. (2022). Creep tests on salt samples performed at very small stresses. *Proc. SMRI Spring Meeting*, Rapid City, SD, USA.
- Carter, N. L., Horseman, S. T., Russell, J. E., and Handin, J. (1993) Rheology of rocksalt, *J. Struct. Geol.*, 15, 1257–1271, [https://doi.org/10.1016/0191-8141\(93\)90168-A](https://doi.org/10.1016/0191-8141(93)90168-A).
- Charpentier, J.P., Bérest, P., Blum, P.A. (1999). Creep of rock salt under small loading. *Proc. 5th Conf. Mech. Beh. Salt*, Bucharest, Romania, Cristescu D. Hardy R.H Jr, Simionescu O.(eds), Balkema, 135-140.

Desbois, G., Urai, J.L., Schmatz, J., Zavada, P., de Bresser, H. (2012) The distribution of fluids in natural rock salt to understand deformation mechanisms. *Mechanical Behavior of Salt VII – Bérest, Ghoreychi, Hadj-Hassen & Tijani (eds) 2012 Taylor & Francis Group, London, ISBN 978-0-415-62122-9*

Schenk, O., Urai, J.L. (2004) Microstructural evolution and grain boundary structure during static recrystallization in synthetic polycrystals of Sodium Chloride containing saturated brine. *Contrib Mineral Petrol* 146, 671–682 (2004). <https://doi.org/10.1007/s00410-003-0522-6>

Schléder, Z. and Urai, J. L.(2005) Microstructural evolution of deformation-modified primary halite from the Middle Triassic Röt Formation at Hengelo, The Netherlands, *Int J Earth Sci (Geol Rundsch)*, 94, 941–955, <https://doi.org/10.1007/s00531-005-0503-2>.

Spiers C.J., Schutjens P.M.T.M., Brzesowsky R.H., Peach C.J., Liezenberg J.L., Zwart H.J. (1990). Experimental determination of the constitutive parameters governing creep of rocksalt by pressure solution. *Geological Society Special Publication 54, Deformation Mechanisms, Rheology and Tectonics*, The Geological Society, London, England. 54:215–227. <https://doi.org/10.1144/GSL.SP.1990.054.01.21>

Ter Heege, J.H., De Bresser, J.H.P. and Spiers, C.J. (2005). Rheological behaviour of synthetic rocksalt: The interplay between water, dynamic recrystallization and deformation mechanisms. *J. Struct. Geol.* 27 (6), 948-963.

Urai, J.L., Spiers, C.J., Zwart, H.J. & Lister, G.S. (1986). Weakening of rock salt by water during long-term creep. *Nature* 324 (6097), 554-557.

Urai J.L., Spiers C.J. (2007). The effect of grain boundary water on deformation mechanisms and rheology of rocksalt during long-term deformation. In: Wallner M, Lux K, Minkley W, Hardy, Jr. H (eds) *Proceedings of the sixth conference on the mechanical behavior of salt*. A. A. Balkema, Netherlands, 149–158.

Watanabe, T. , Peach, C. J. (2002) Electrical impedance measurement of plastically deforming halite rocks at 125°C and 50 MPa, *J. Geophys. Res.*, 107(B1), doi:10.1029/2001JB000204.

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APPENDIX

ZUIDWENDING WP2.3 Small-Deviatoric-Stress Creep Tests in the Mine

APPENDIX
by MaP
09.03.2024

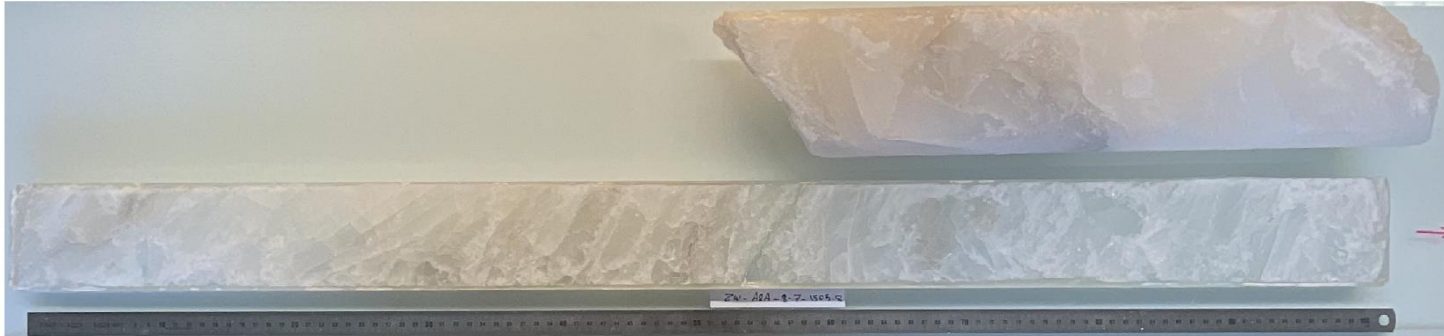
Overview

					Load Mine [MPa]				grain size initial twin (dmed) [mm]		grain size after mine test (dmed) [mm]	
Well information	depth [m]	MaP ID	IfG ID	Mine ID	start	14.03.2023	comment	twin ID	dmed [mm]	Std_deviation	dmed [mm]	Std_deviation
ZW_A7B_1_13	1401,18	ZW_MaP_40	TC2	Zuidwending 2	0,6	0,60		ZW_MaP_05	1,05	2,63	0,46	2,85
ZW_A7B_1_13	1401,38	ZW_MaP_41	TC3	Zuidwending 4	0,6	0,60	pre-deformed	ZW_MaP_05	1,05	2,63	0,30	2,63
ZW_A7B_1_13	1401,58	ZW_MaP_42	TC4	Zuidwending 3	3,0	1,50		ZW_MaP_05	1,05	2,63	1,61	2,64
ZW A2A_8_7	1505,9	ZW_MaP_43	TC5	Zuidwending 1	0,6	0,60		ZW_MaP_04	1,06	2,76	0,33	2,94

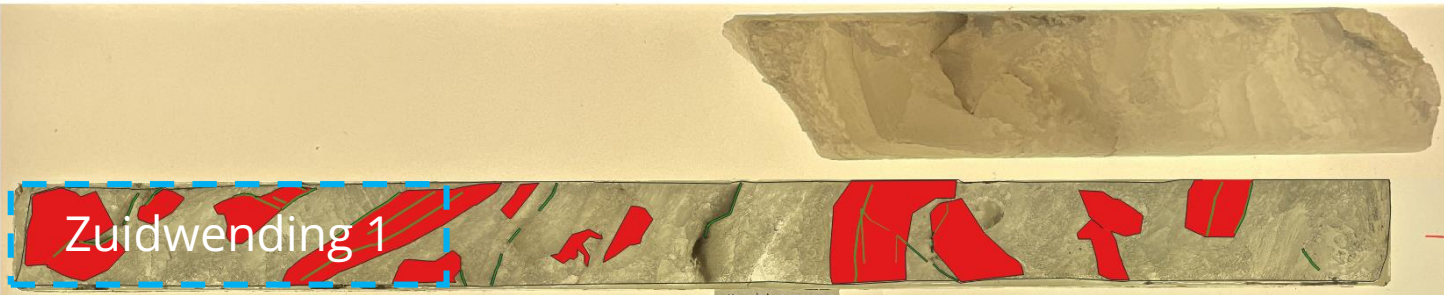
Sample selection criteria

Coarse-grained, clear salt

ZW_MaP_43	TC5	Zuidwending 1	0,6 MPa
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Anhydrite = 1.5%
Megagrain = 28.9%
Rest = 69.6%



Fine-grained, clear salt with mega-grain

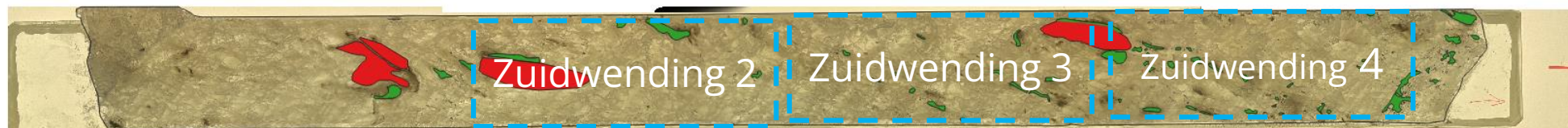
ZW_MaP_40	TC2	Zuidwending 2	0,6 MPa
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Fine-grained, clear salt

ZW_MaP_42	TC4	Zuidwending 3	3,0 MPa
-----------	-----	---------------	---------

Fine-grained, clear salt, pre-deformed to 20% axial strain → diminished grain size

ZW_MaP_41	TC3	Zuidwending 4	Pre-def, 0,6 MPa
-----------	-----	---------------	------------------



Anhydrite = 1.9%
Megagrain = 3.7%
Rest = 94.4%

ZW2.3 sampling procedure

In the Altaussee mine



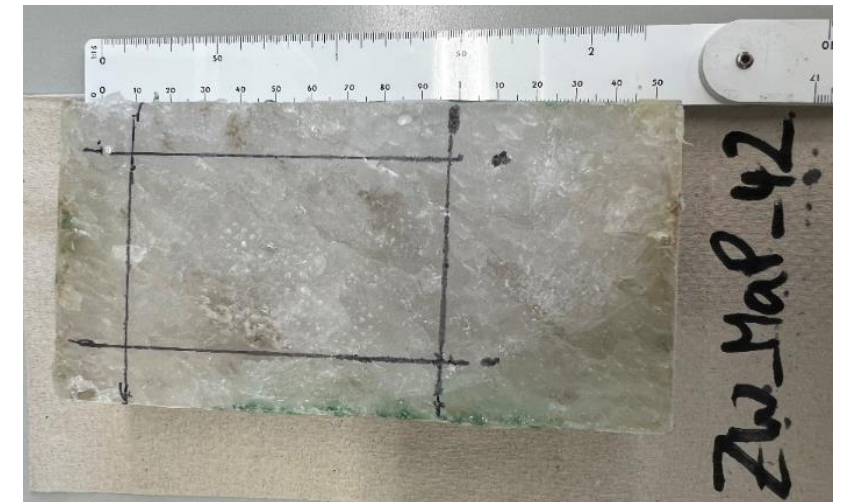
Preparation in Aachen



Stabilisation core piece



Slab with location for thin section



ZW_MaP_43

TC5

Zuidwending 1

0,6 MPa

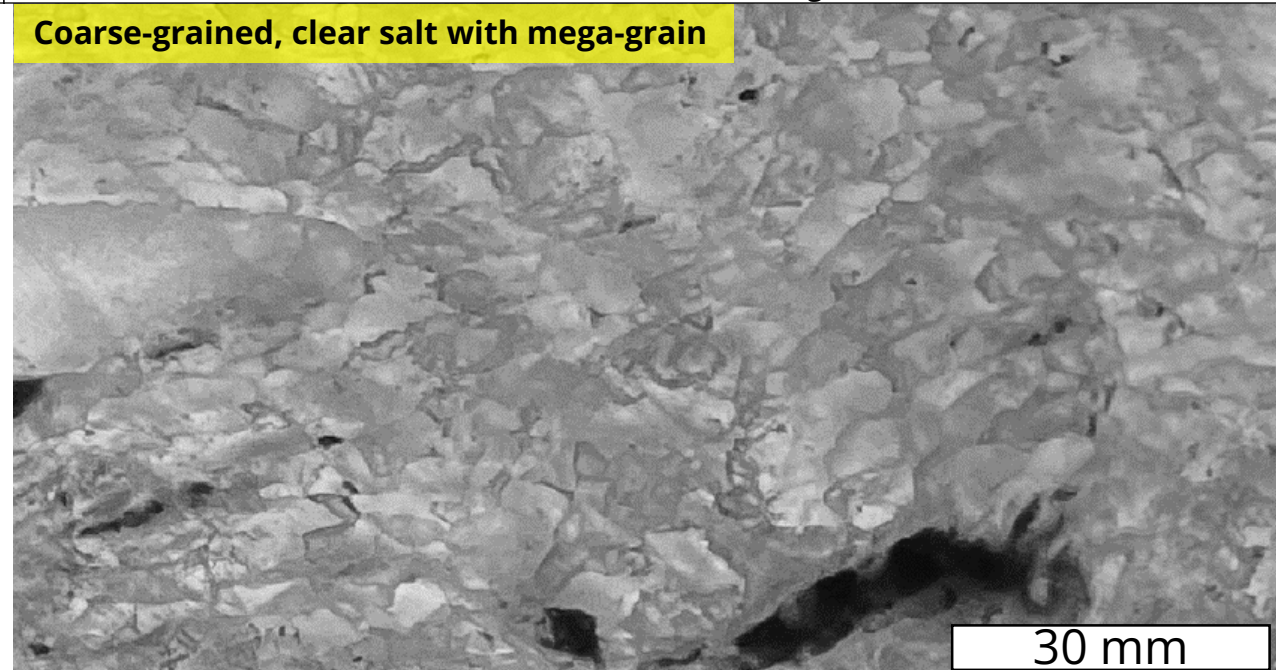
Coarse-grained, clear salt with mega-grain

ZW_MaP_40

TC2

Zuidwending 2

0,6 MPa

Coarse-grained, clear salt with mega-grain

ZW_MaP_42

TC4

Zuidwending 3

3,0 MPa -> 1,5 MPa

Fine-grained, clear salt

ZW_MaP_41

TC3

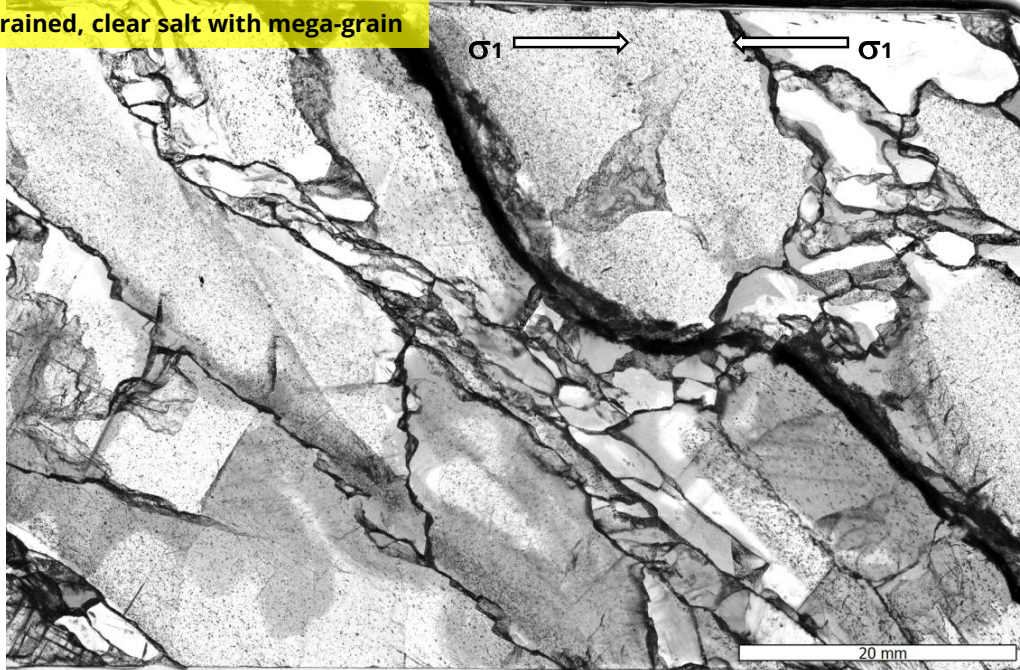
Zuidwending 4

Pre-def, 0,6 MPa

Fine-grained, clear salt, pre-deformed

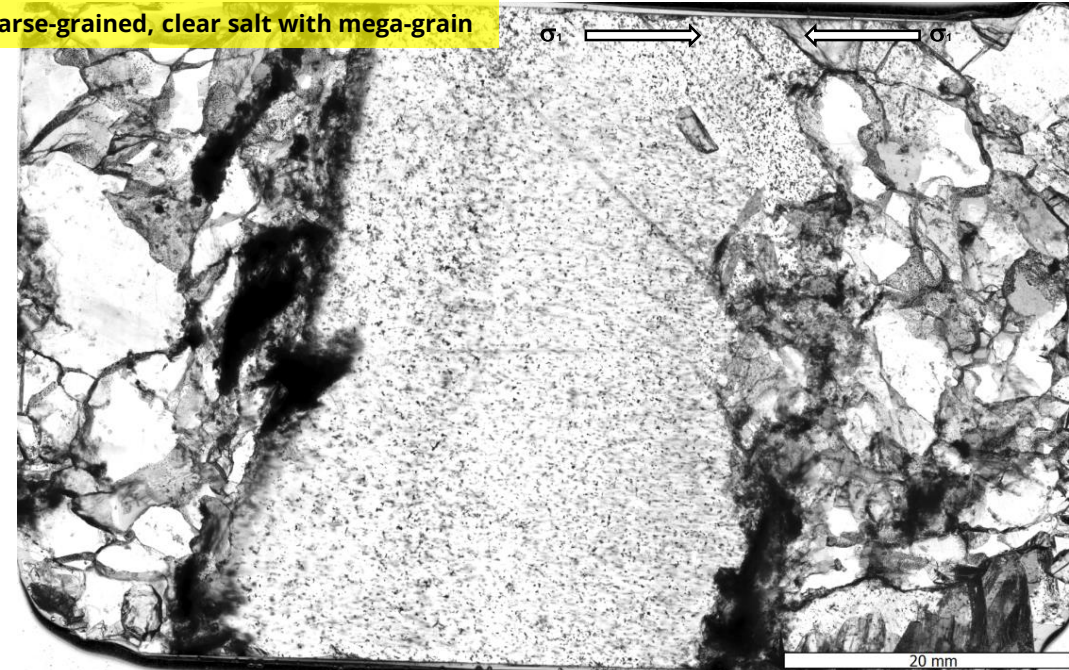
ZW_MaP_43 TC5 Zuidwending 1 0,6 MPa

Coarse-grained, clear salt with mega-grain



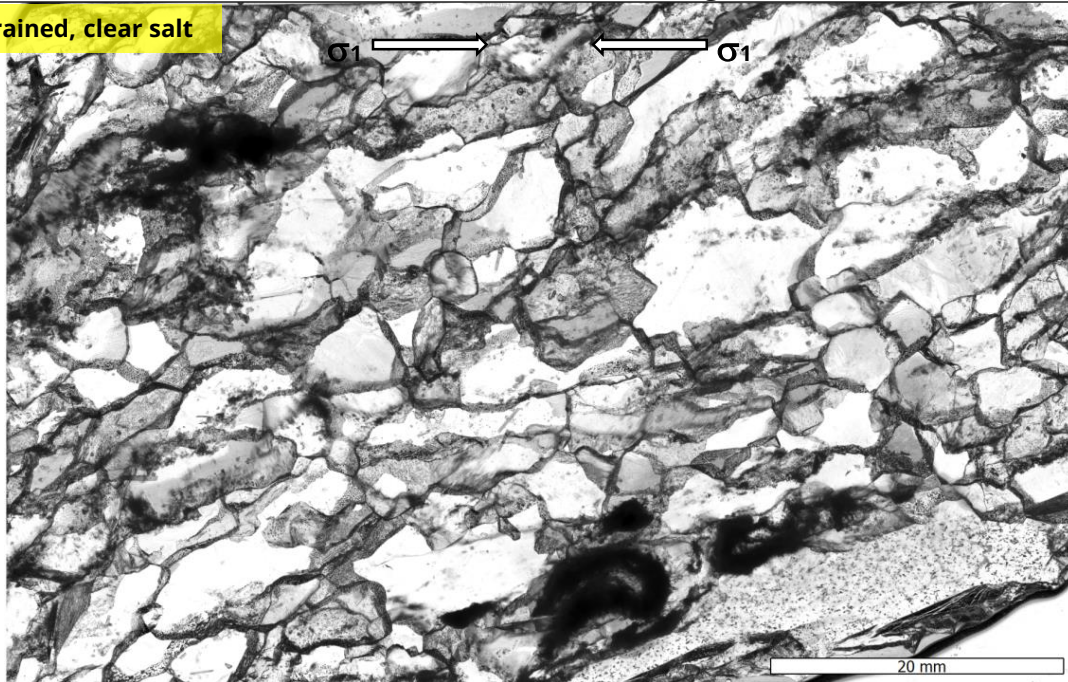
ZW_MaP_40 TC2 Zuidwending 2 0,6 MPa

Coarse-grained, clear salt with mega-grain



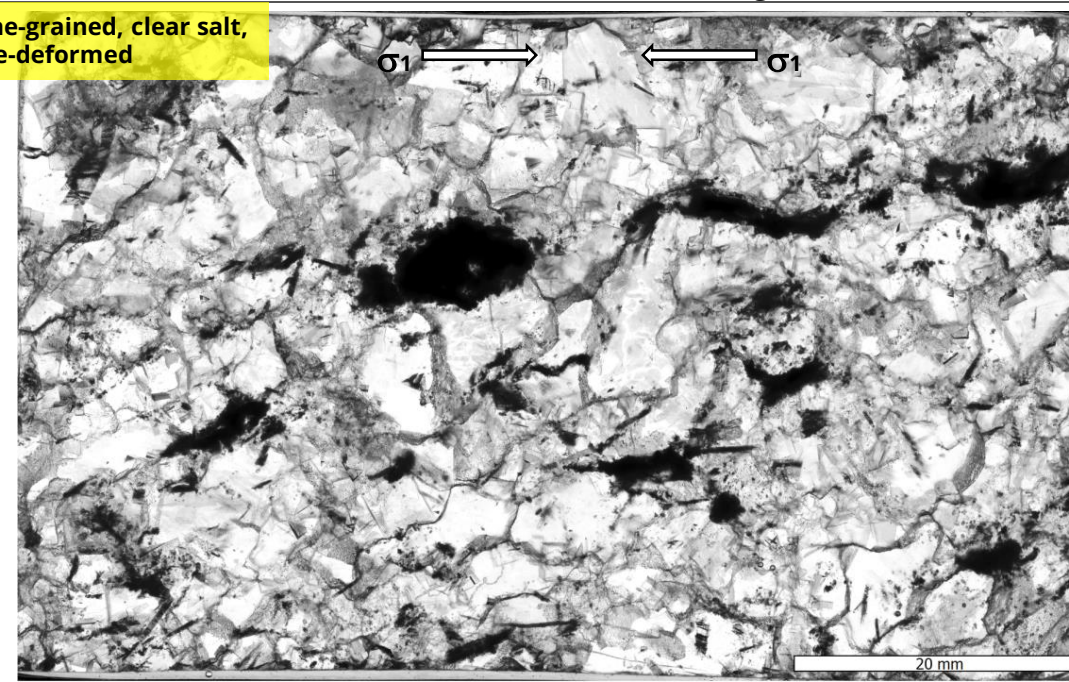
ZW_MaP_42 TC4 Zuidwending 3 3,0 MPa -> 1,5 MPa

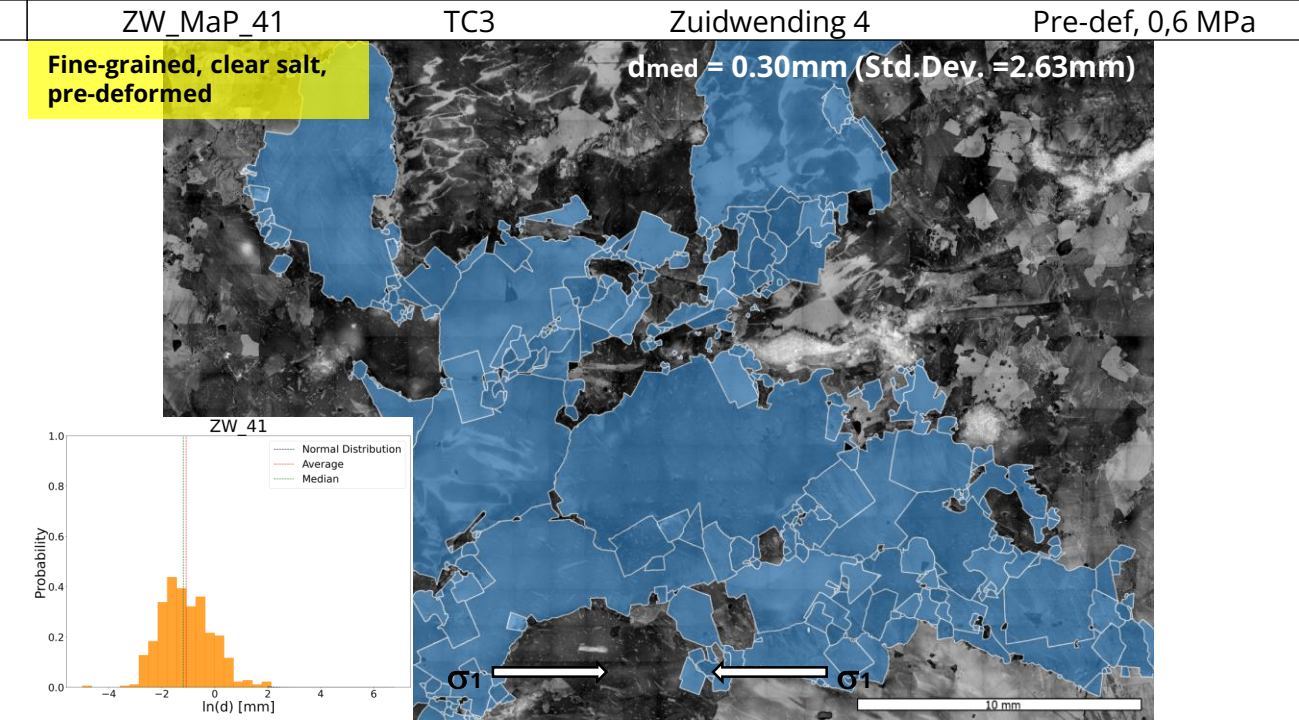
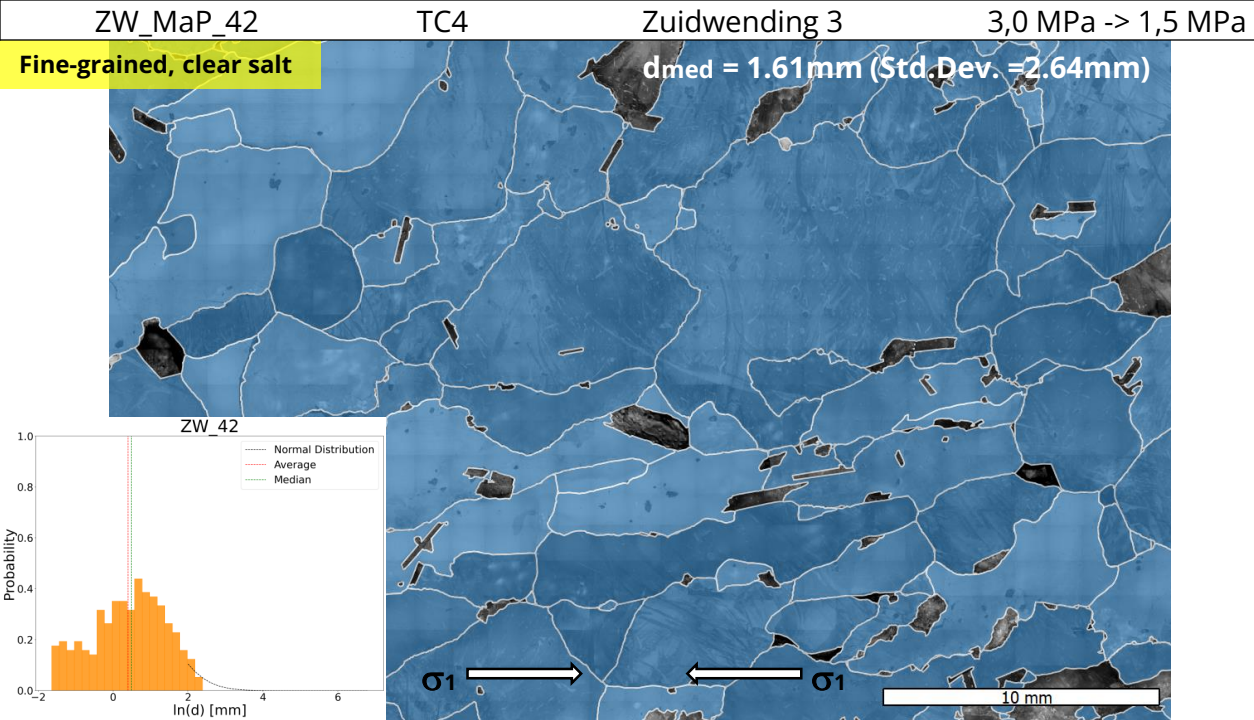
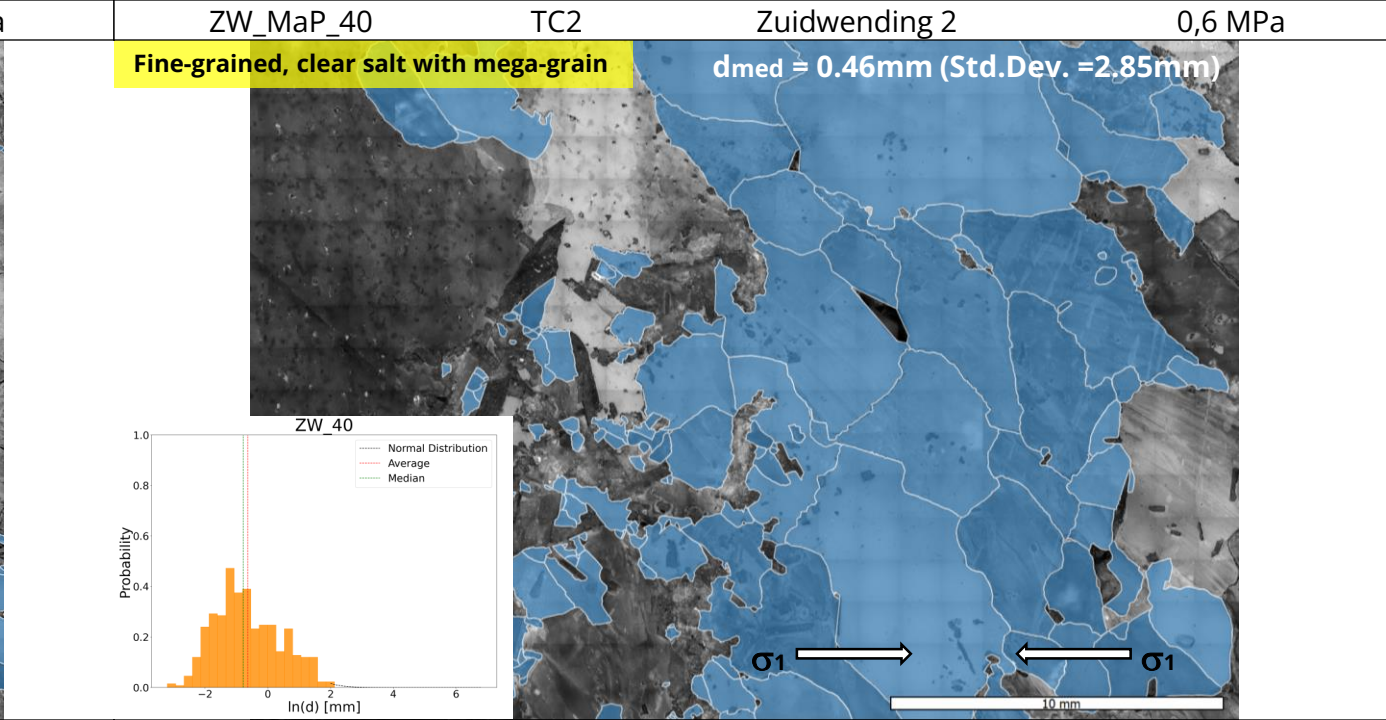
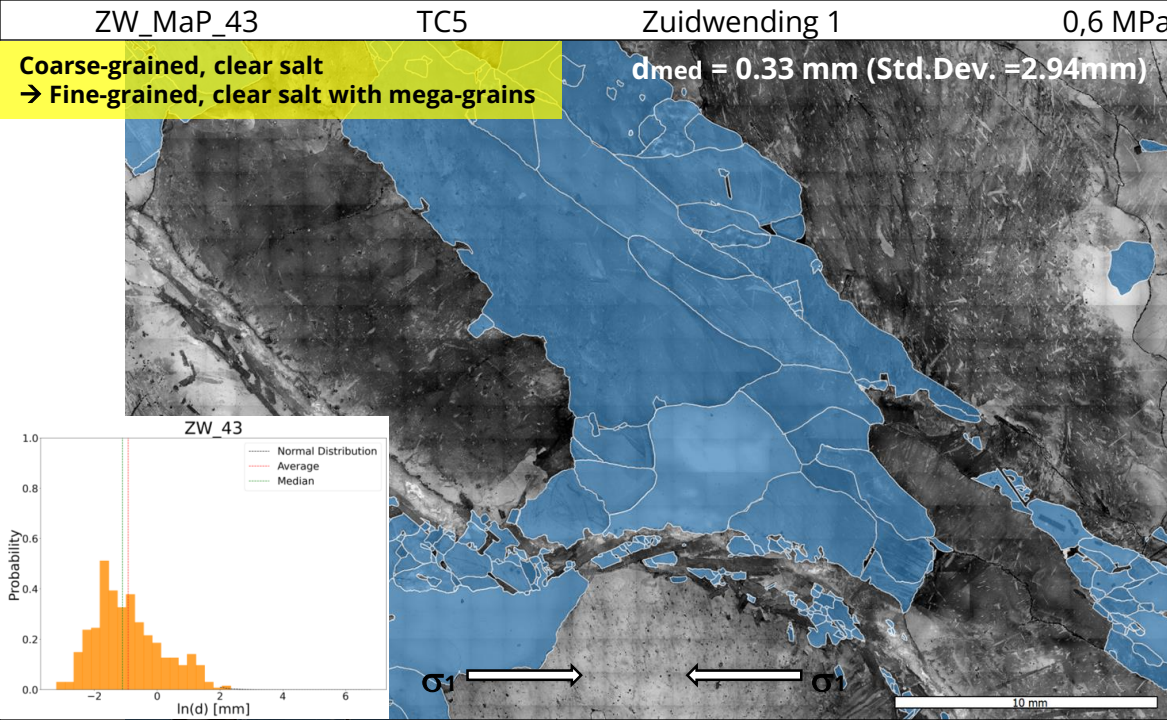
Fine-grained, clear salt



ZW_MaP_41 TC3 Zuidwending 4 Pre-def, 0,6 MPa

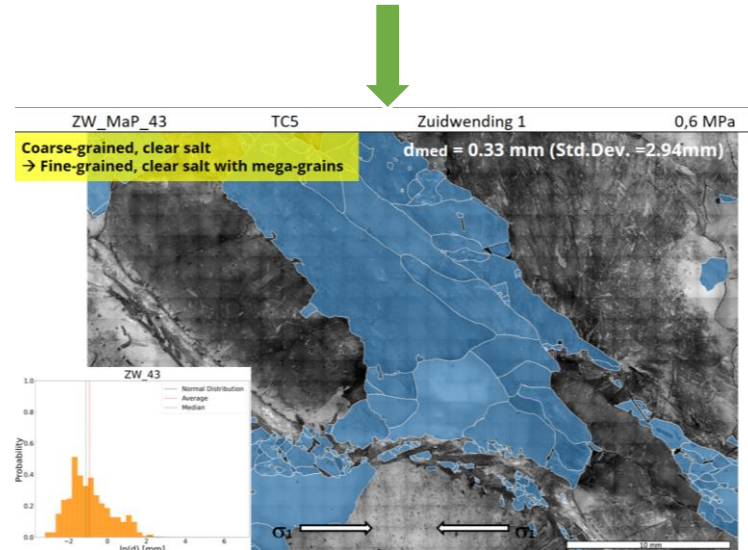
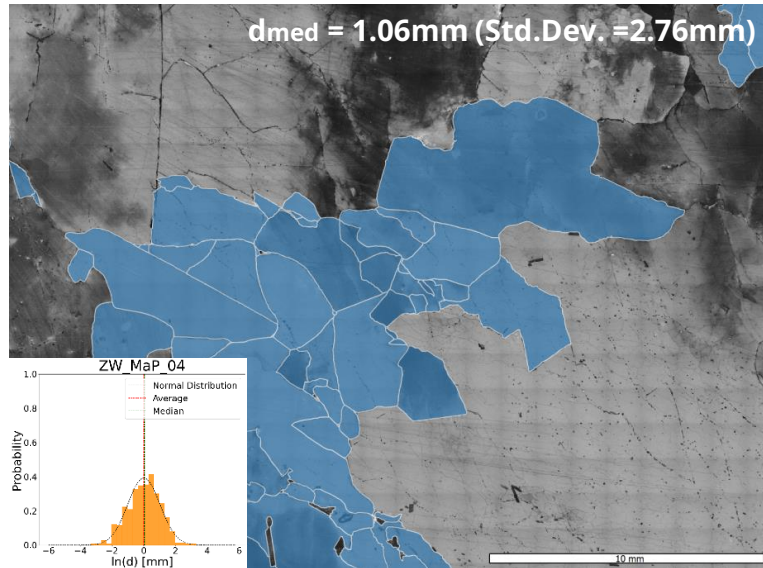
Fine-grained, clear salt, pre-deformed



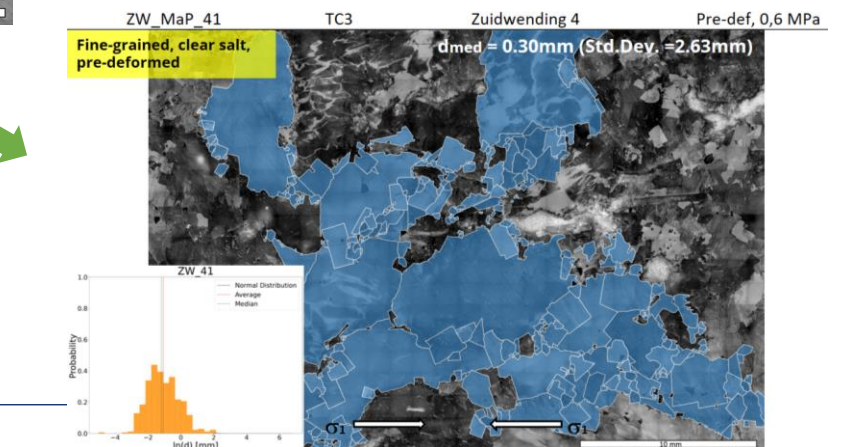
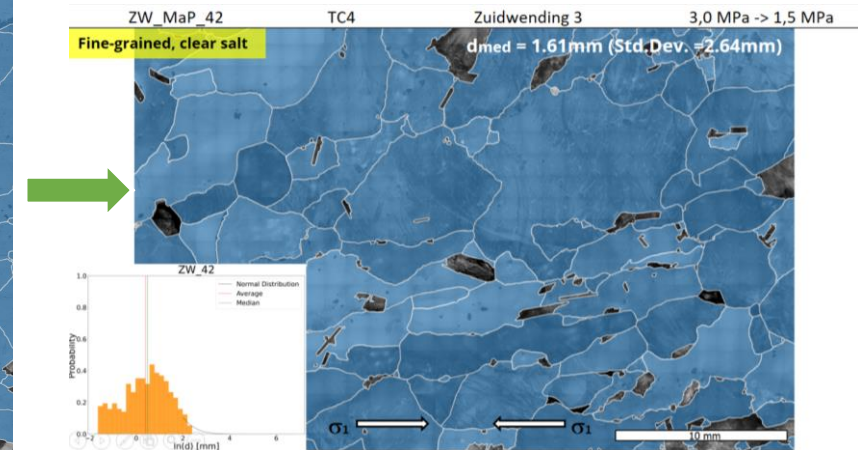
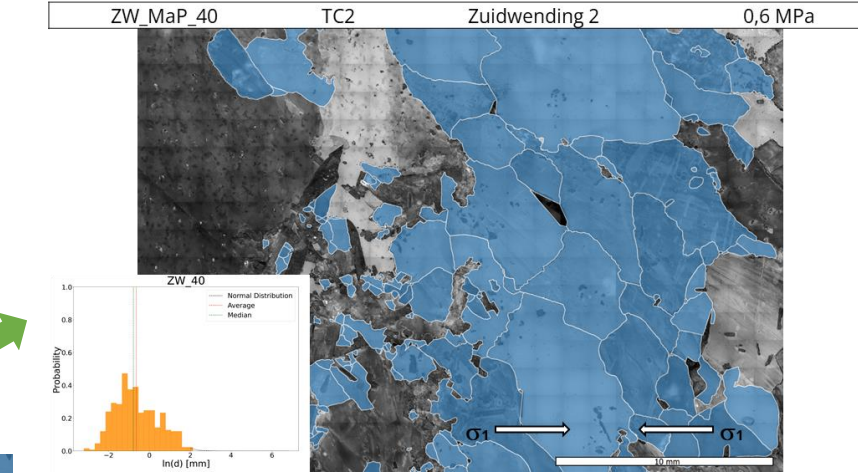
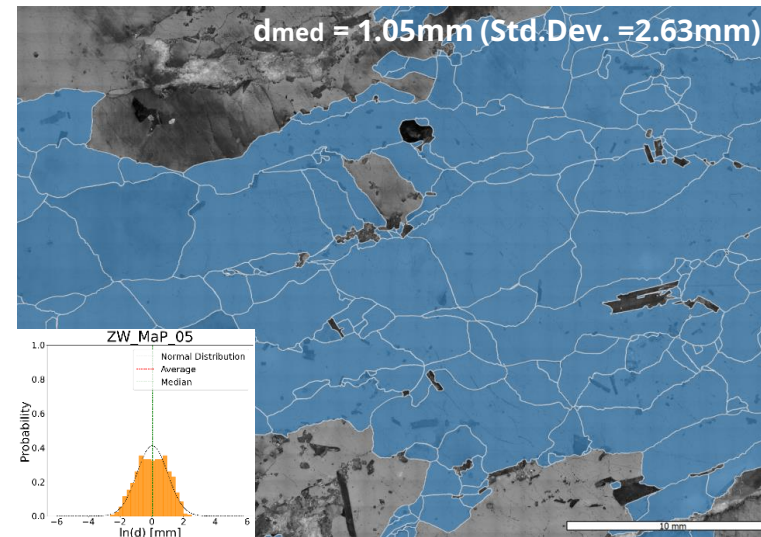


GSD before and after creep-test

Zuidwending 1: Initial twin



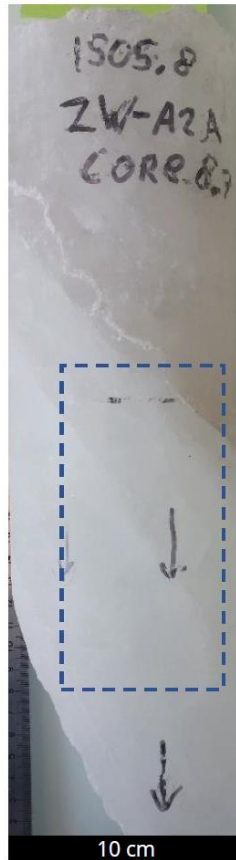
Zuidwending 2,3,4: Initial twin



Zuidwending-1_TC5_MaP_43_0.6MPa

Core piece photography: **Zuidwending-1_TC5_MaP_43_0.6MPa**

Reflected light



Transmitted light



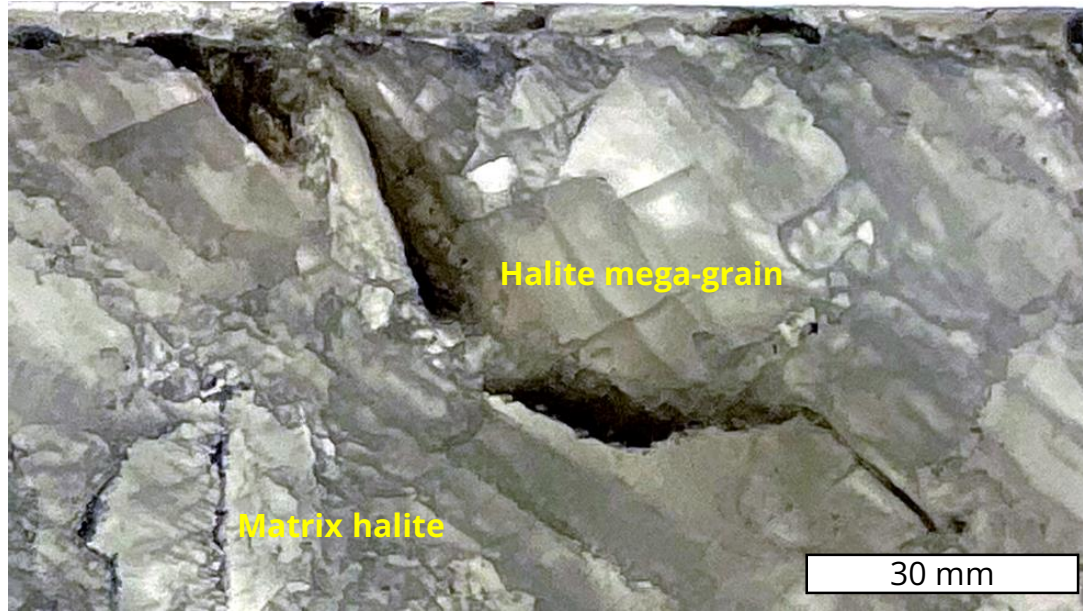
Transmitted light
(cut-side)



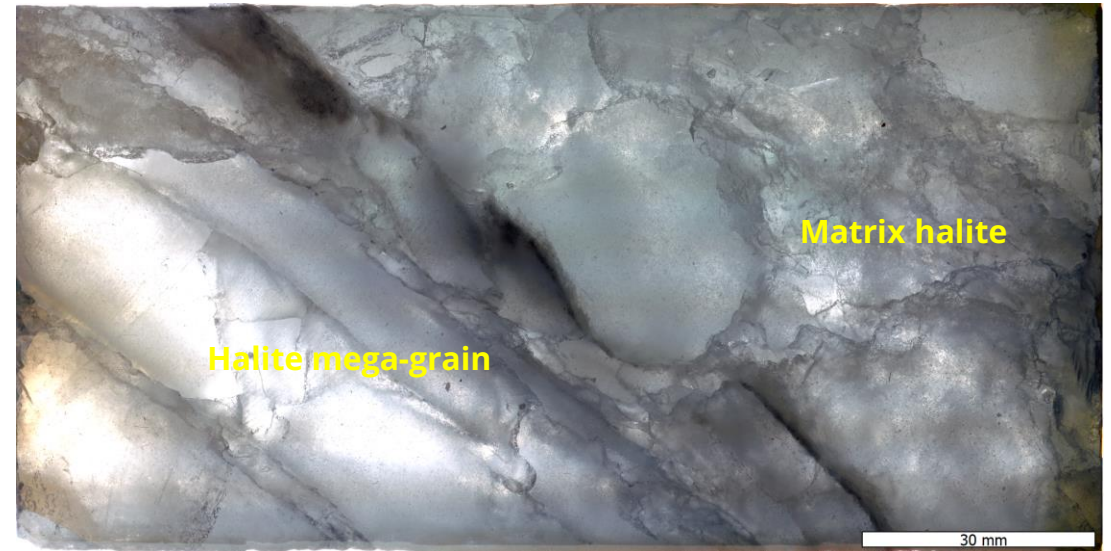
Reflected light
(cut-side)



Core piece slab photo: **Zuidwending-1_TC5_MaP_43_0.6MPa**

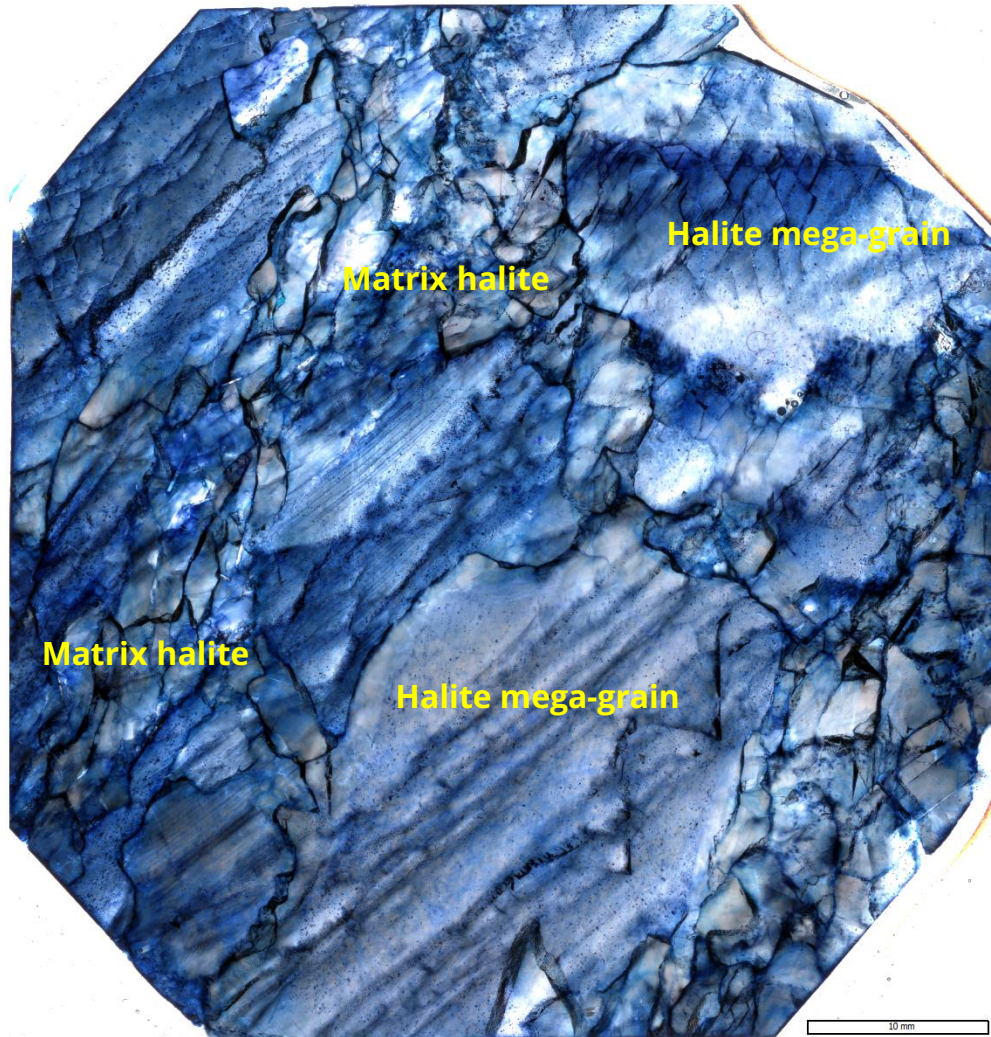


Tectonically-deformed twin: ZW-MaP_04g

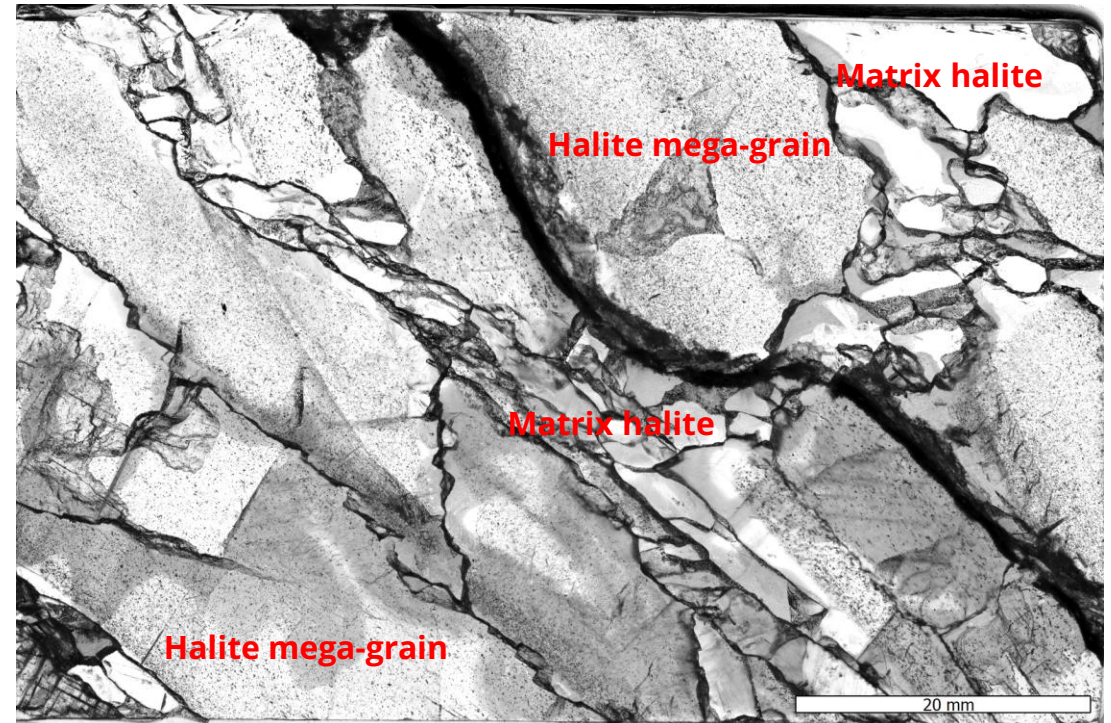


Lab-deformed twin: Zuidwending-1_TC5_MaP_43

Thin section in transmitted light: **Zuidwending-1_TC5_MaP_43_0.6MPa**

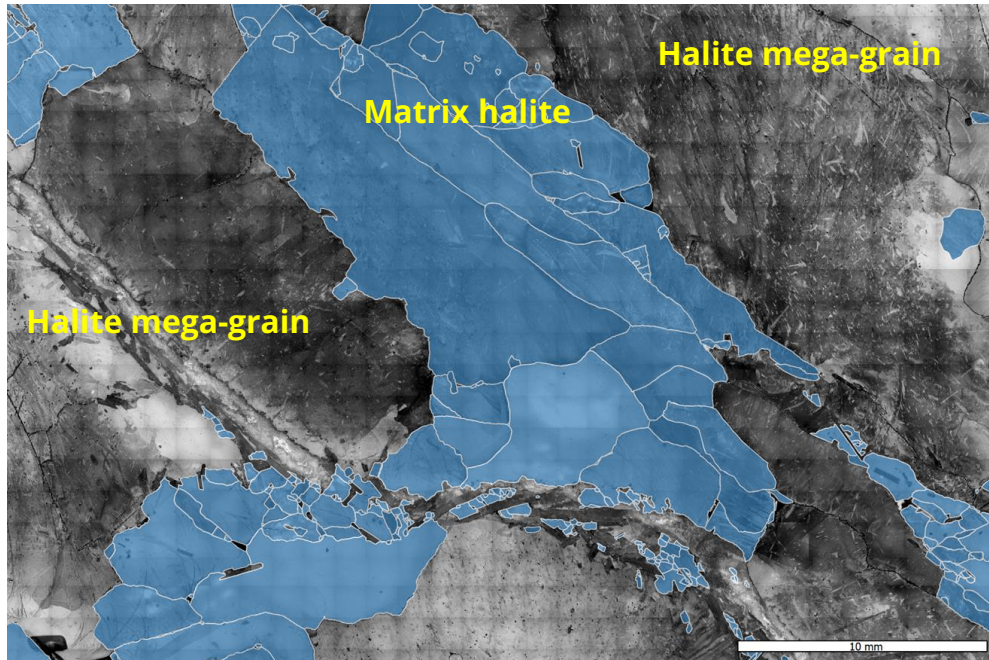


Tectonically-deformed twin: ZW-MaP_04g

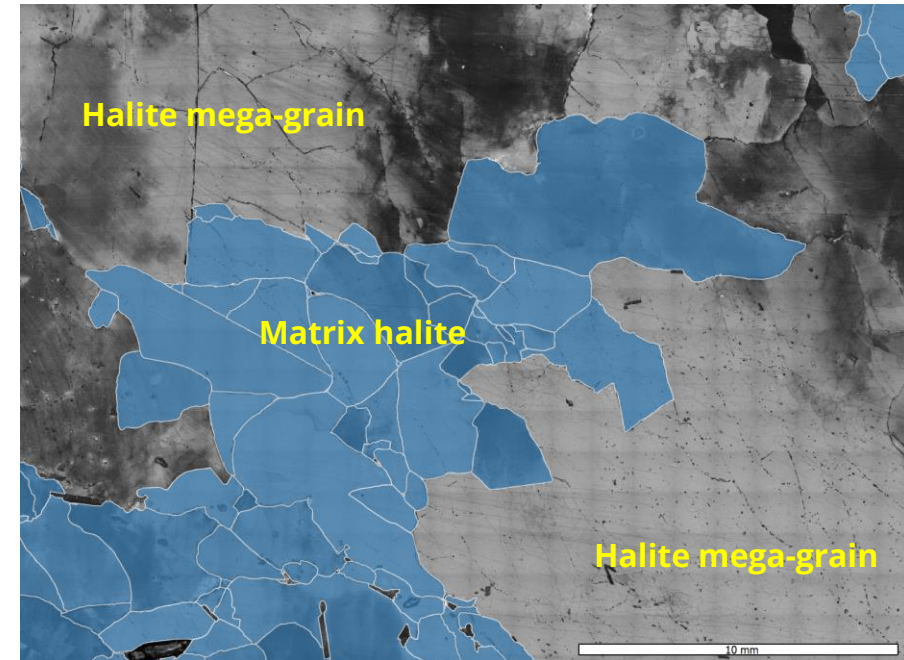


Lab-deformed twin: Zuidwending-1_TC5_MaP_43

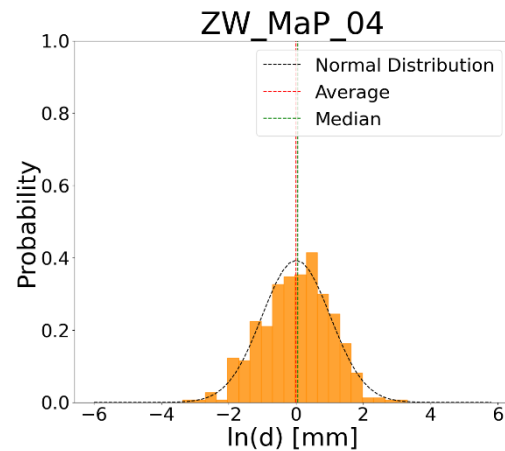
Grain size (d) distribution: **Zuidwending-1_TC5_MaP_43_0.6MPa**



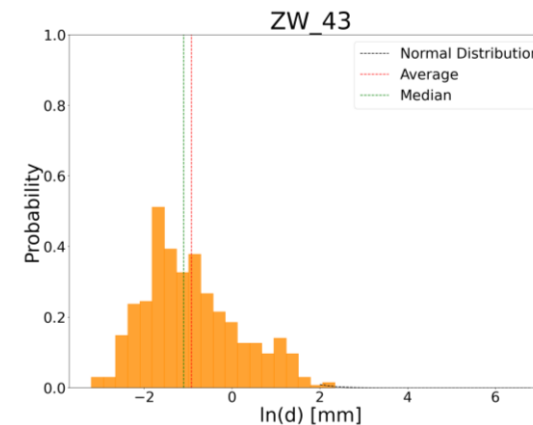
Tectonically-deformed twin: ZW-MaP_04g



Lab-deformed twin: Zuidwending-1_TC5_MaP_43

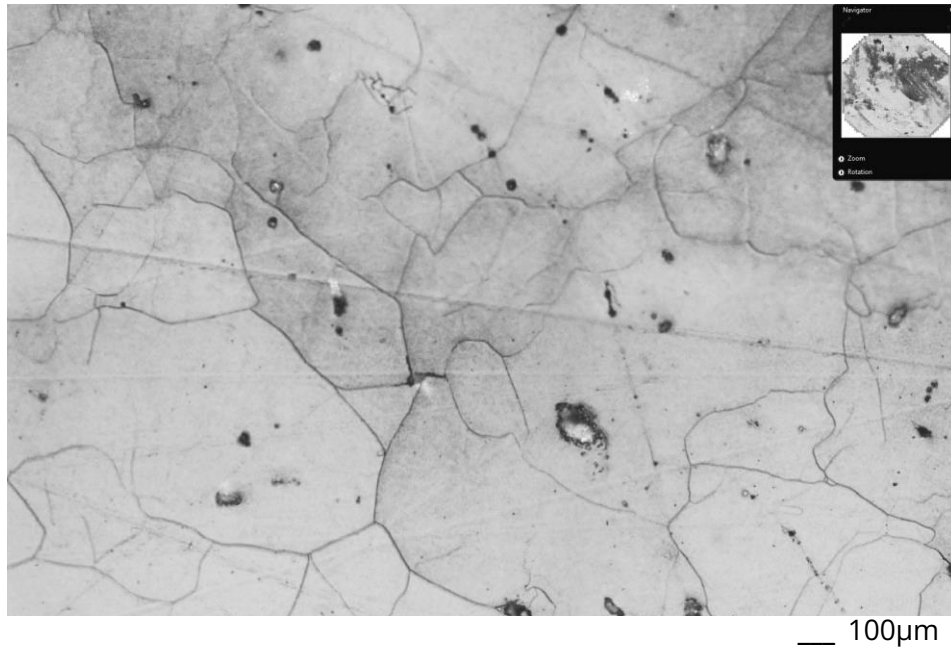


	d [mm]	ln(d)[mm]
Number of grains	440	440
Min [mm]	0.03	-3.37
Max [mm]	27.19	3.30
Median [mm]	1.06	0.06
Average [mm]	1.64	0.00
Std_deviation [mm]	2.12	1.02
Mode [mm]	2.82	1.04

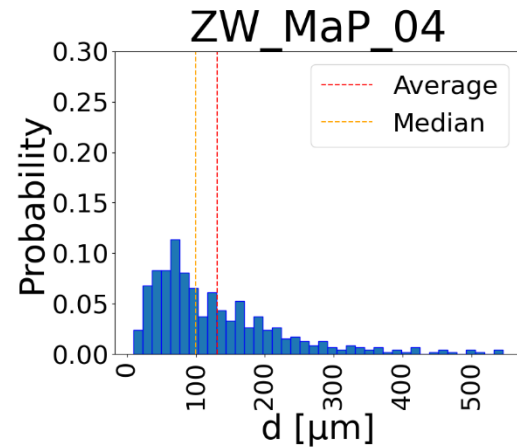


	d [mm]	ln(d)[mm]
Number of grains	501	501
Min [mm]	0.04	-3.21
Max [mm]	10.43	2.34
Median [mm]	0.34	-1.07
Average [mm]	0.76	-0.92
Std_deviation [mm]	1.14	1.07
Mode [mm]	0.19	-1.67

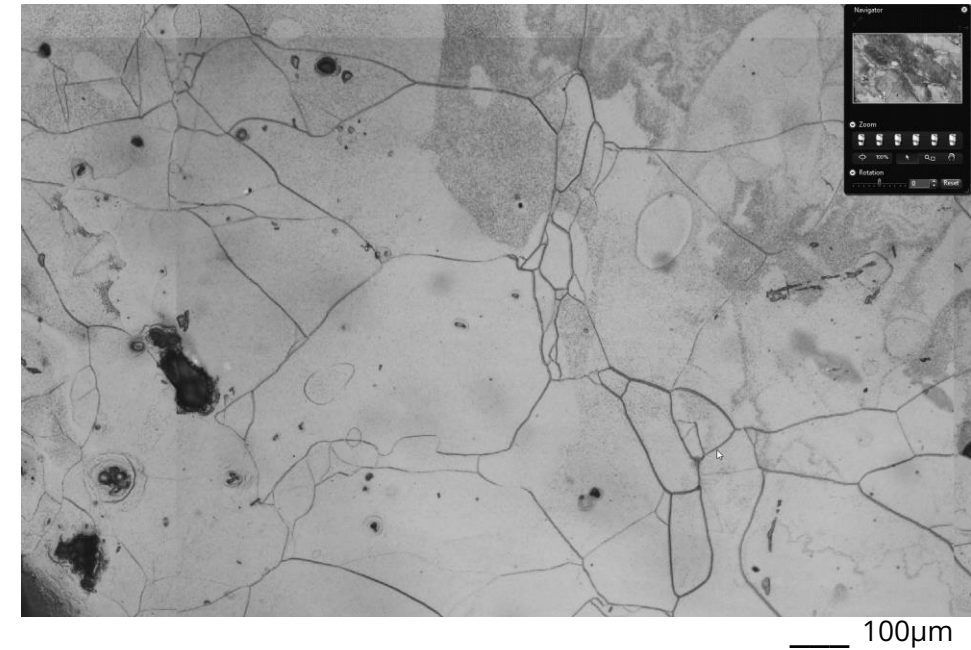
Subgrain size (D) distribution: **Zuidwending-1_TC5_MaP_43_0.6MPa**



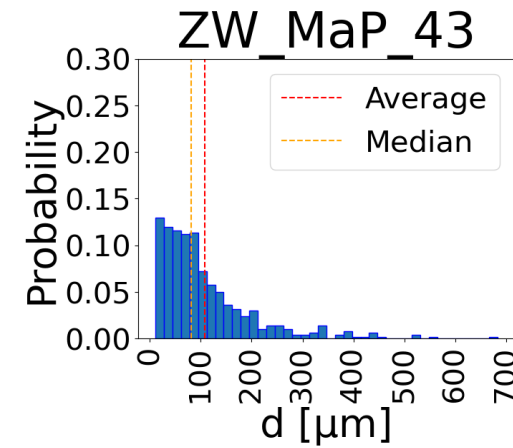
Tectonically-deformed twin: ZW-MaP_04g



	d [µm]	ln(d)[µm]
Number of grains	457	457
Min [µm]	9.14	2.21
Max [µm]	545.47	6.30
Median [µm]	99.57	4.60
Average [µm]	130.42	4.61
Std_deviation [µm]	98.46	0.74
Mode [µm]	58.22	4.06

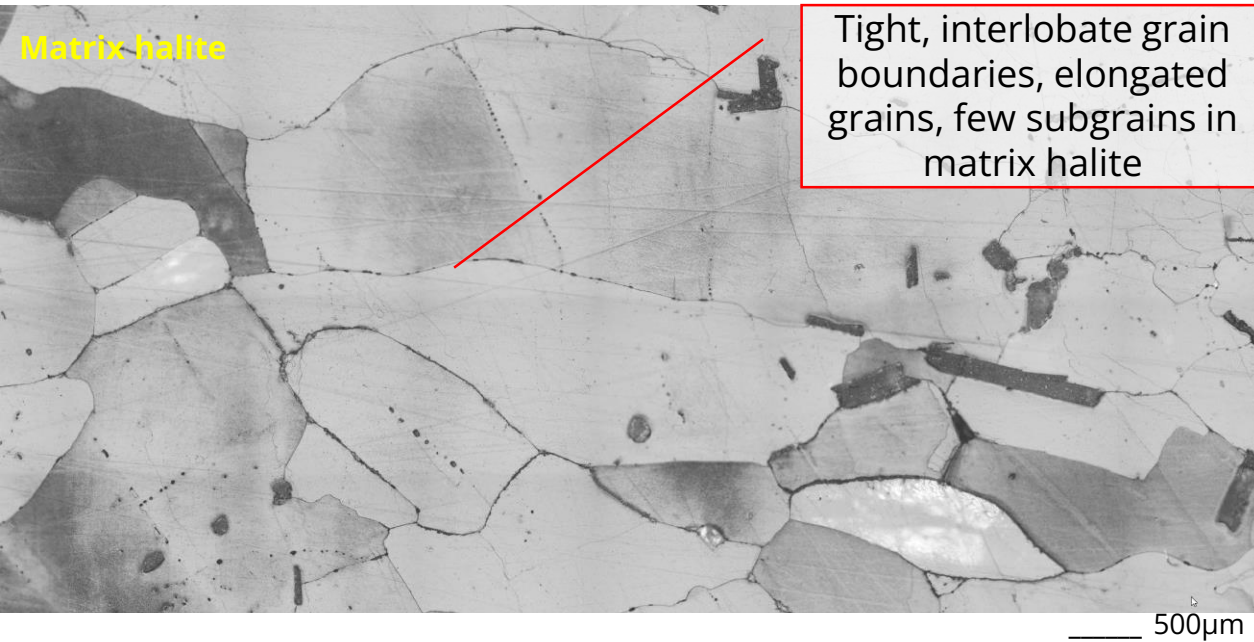


Lab-deformed twin: Zuidwending-1_TC5_MaP_43

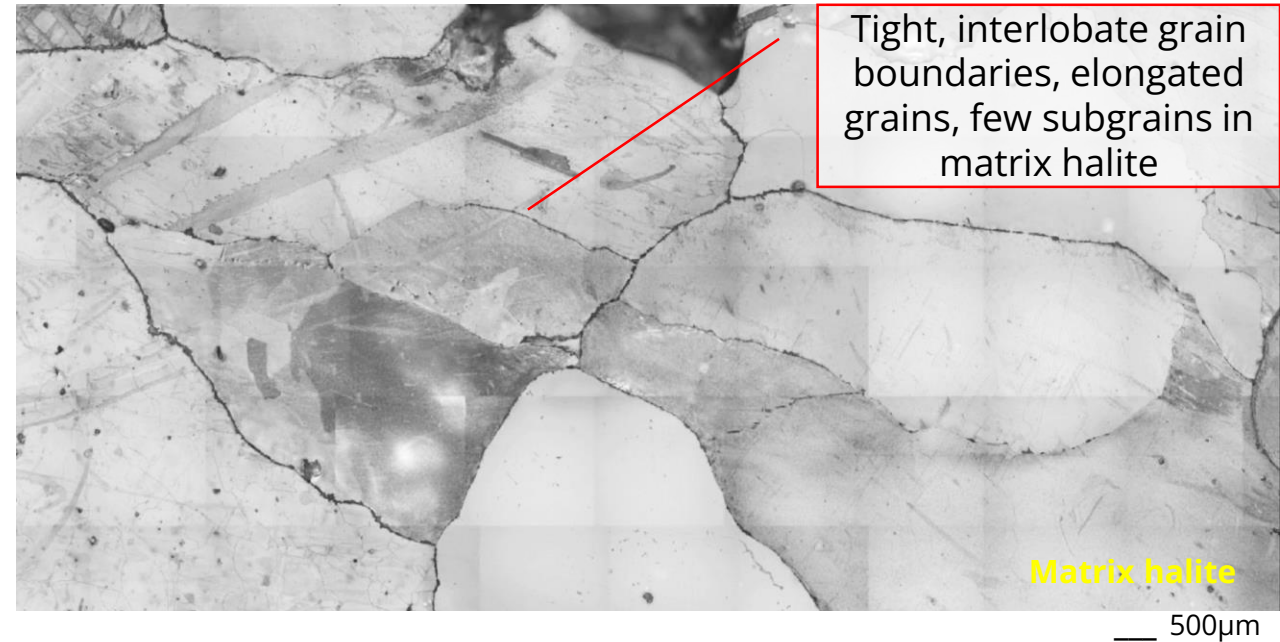


	d [µm]	ln(d)[µm]
Number of grains	501	501
Min [µm]	10.76	2.38
Max [µm]	682.55	6.53
Median [µm]	81.57	4.40
Average [µm]	108.21	4.35
Std_deviation [µm]	94.98	0.83
Mode [µm]	14.41	2.67

Texture: Zuidwending-1_TC5_MaP_43_0.6MPa

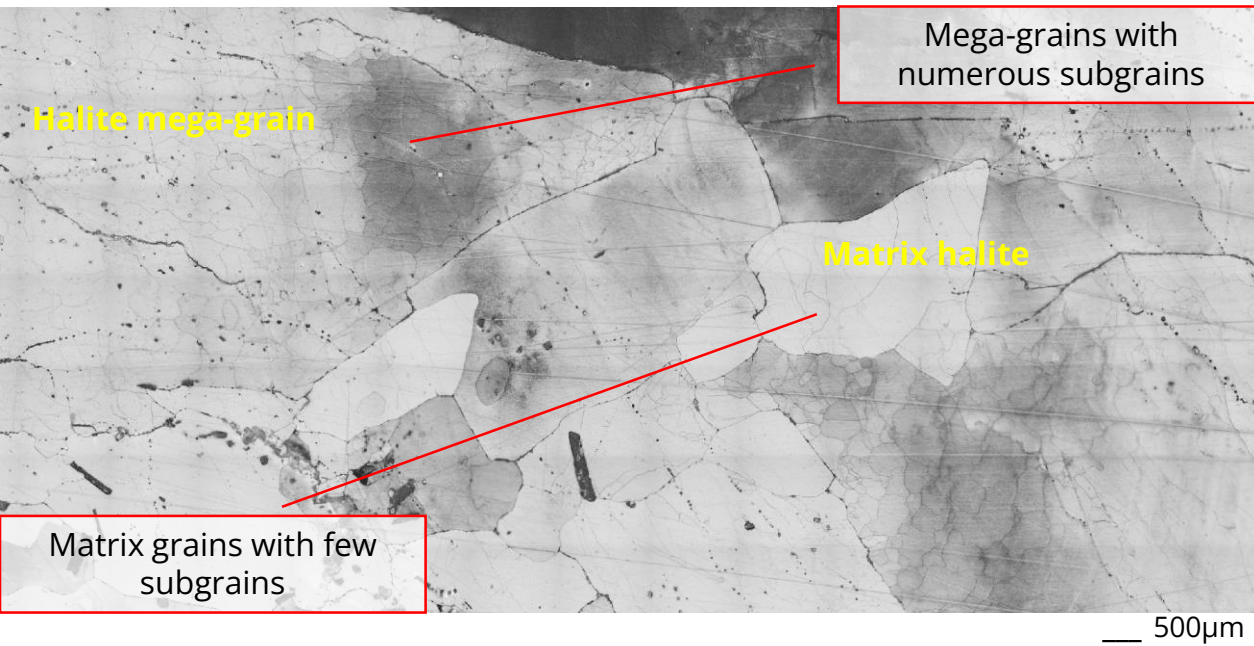


Tectonically-deformed twin: ZW-MaP_04g

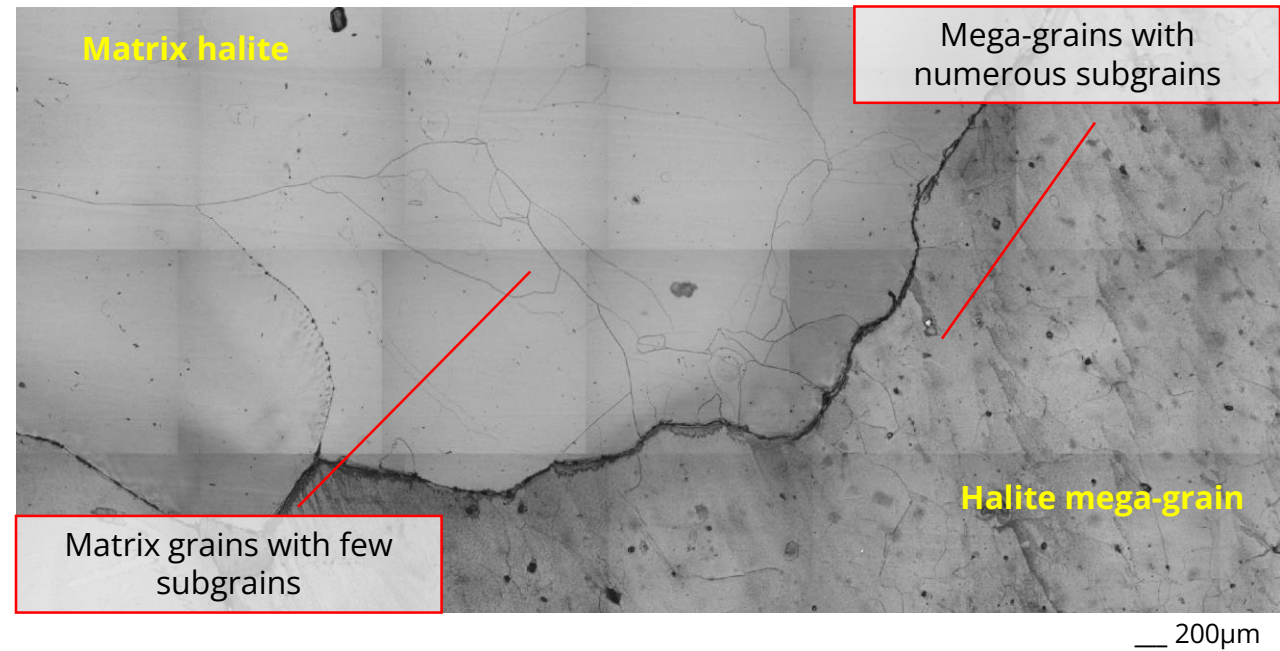


Lab-deformed twin: Zuidwending-1_TC5_MaP_43

Texture: Zuidwending-1_TC5_MaP_43_0.6MPa

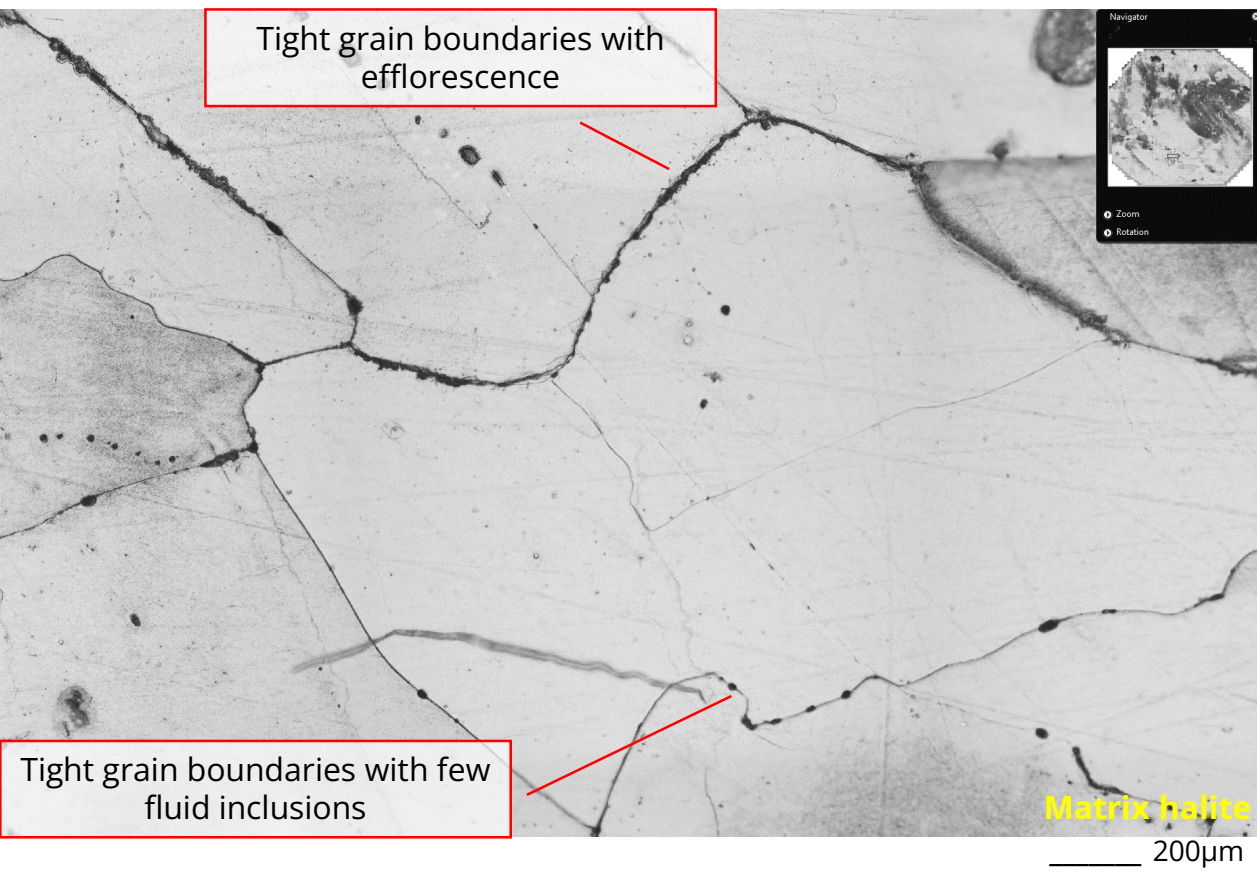


Tectonically-deformed twin: ZW-MaP_04g

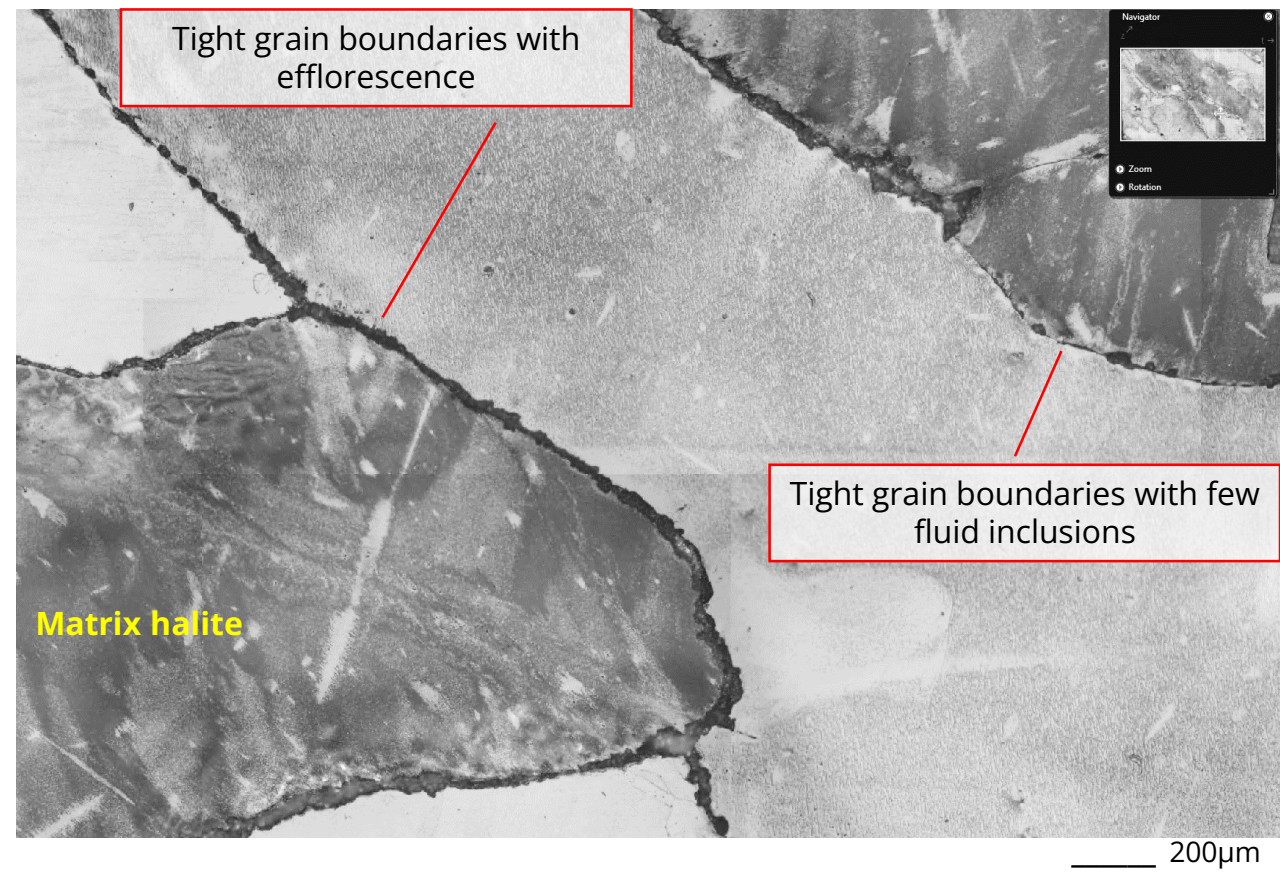


Lab-deformed twin: Zuidwending-1_TC5_MaP_43

Grain boundary structure: Zuidwending-1_TC5_MaP_43_0.6MPa

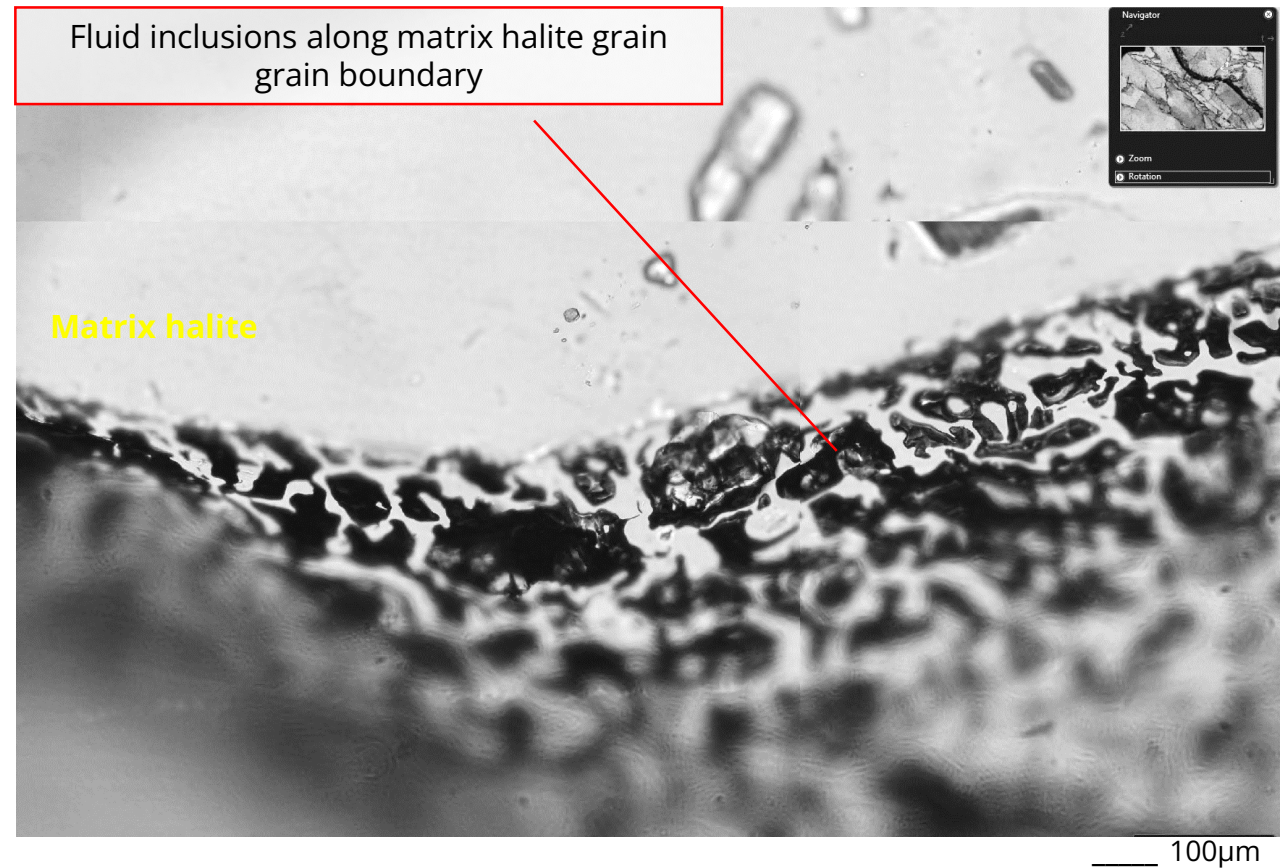
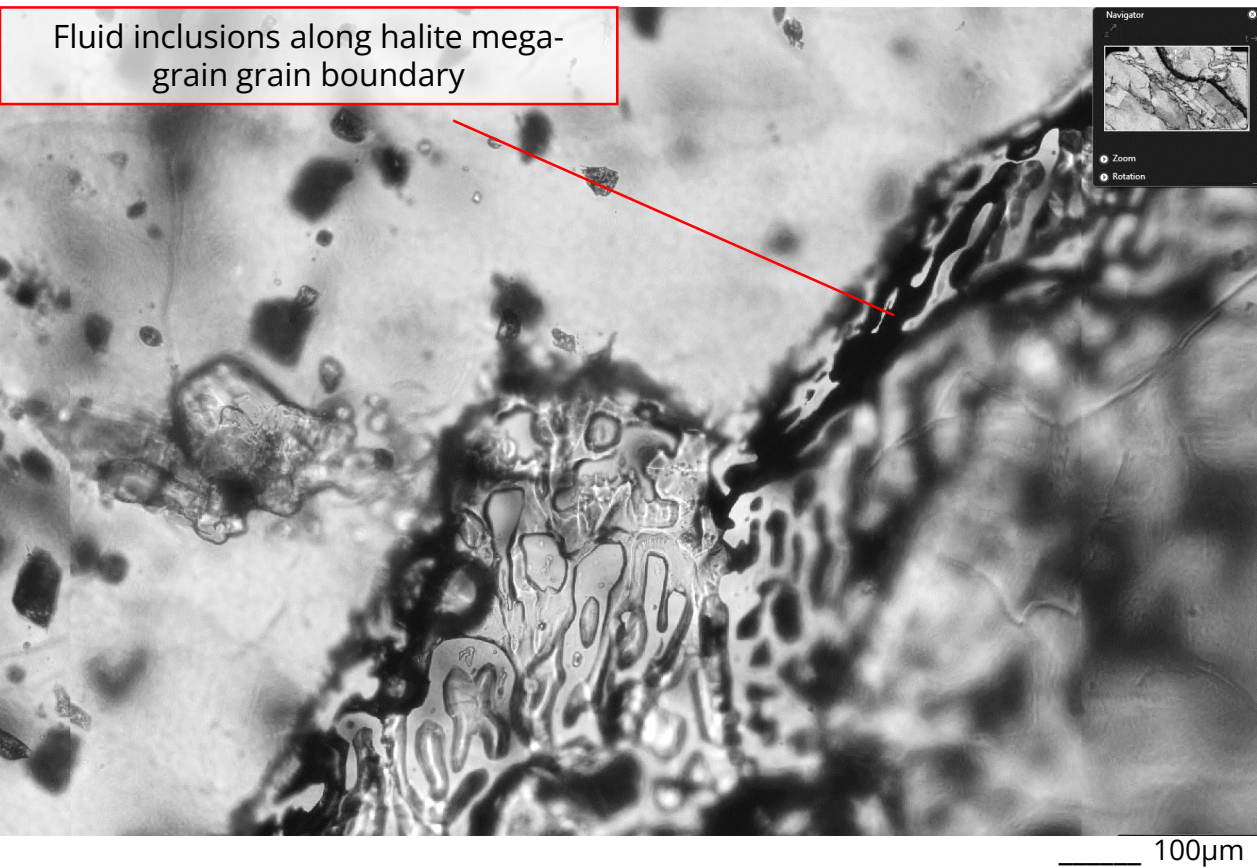


Tectonically-deformed twin: ZW-MaP_04g

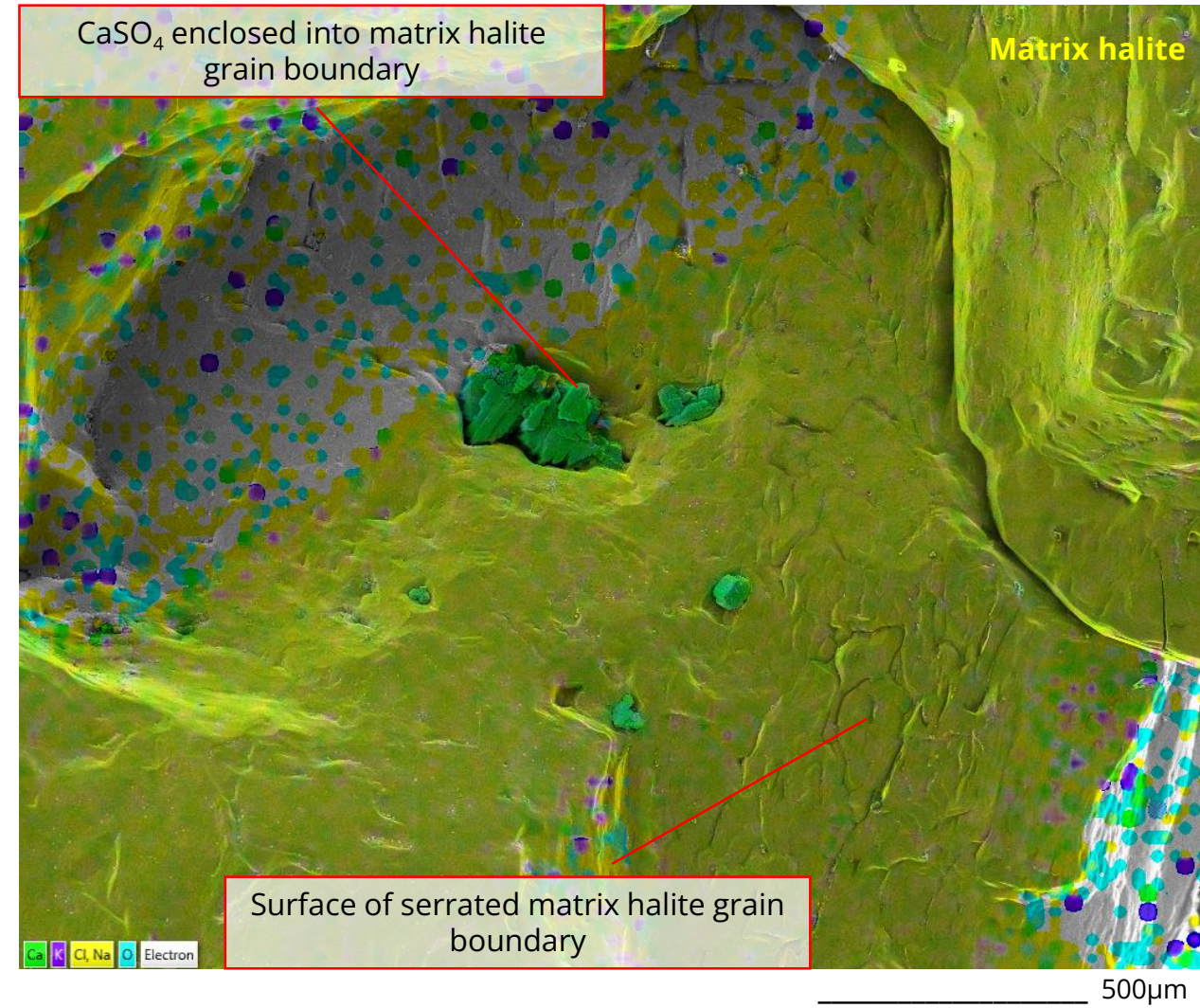
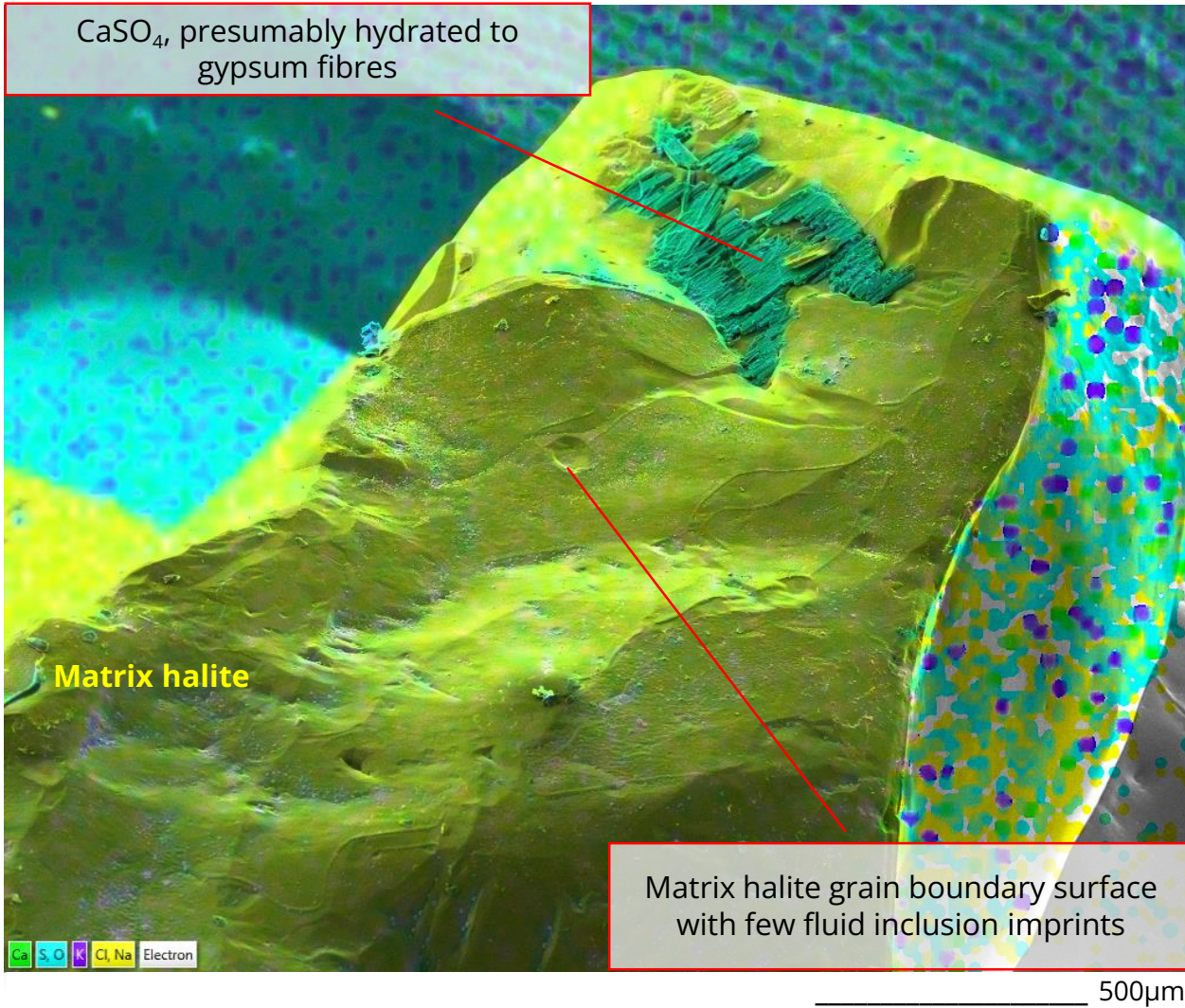


Lab-deformed twin: Zuidwending-1_TC5_MaP_43

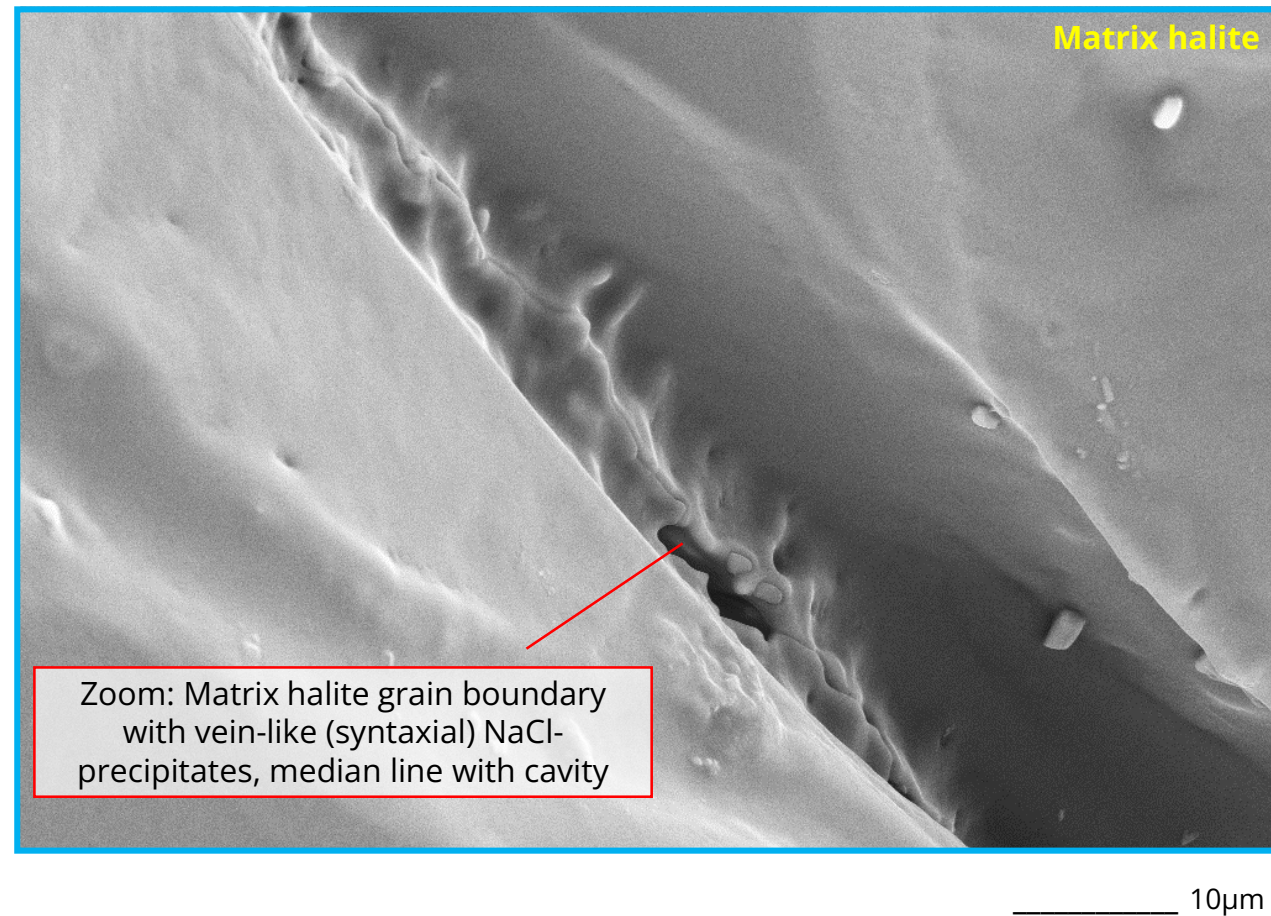
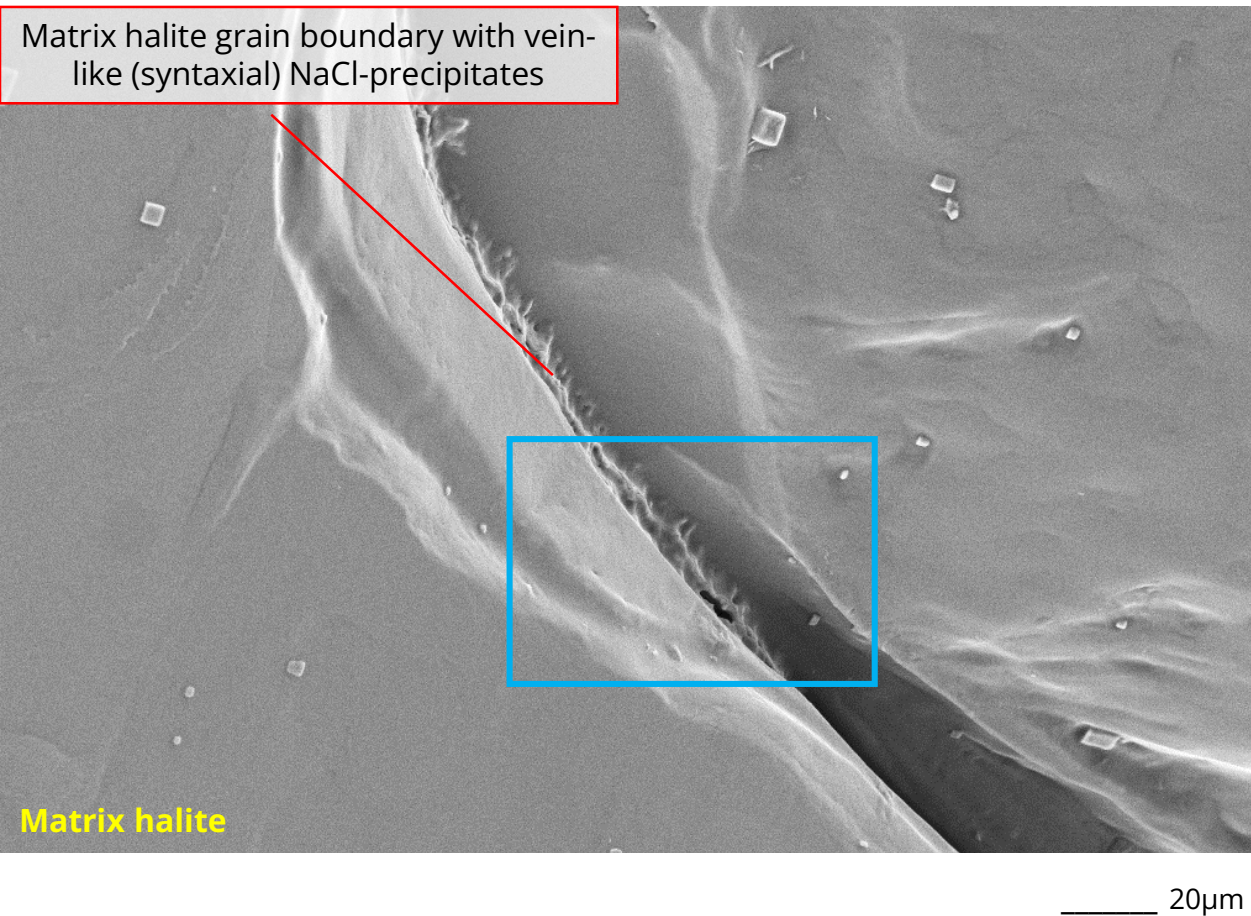
Grain boundary structure: **Zuidwending-1_TC5_MaP_43_0.6MPa**



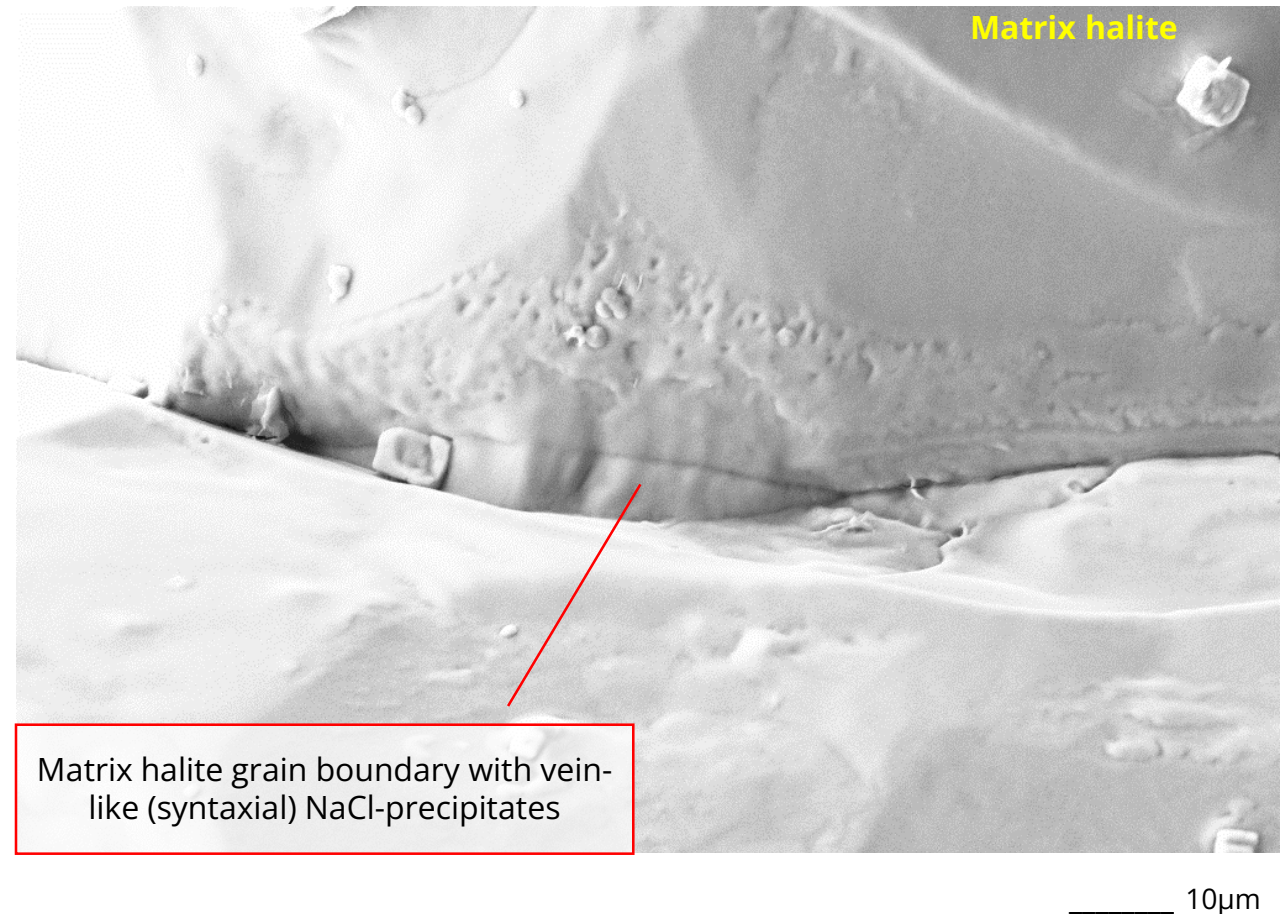
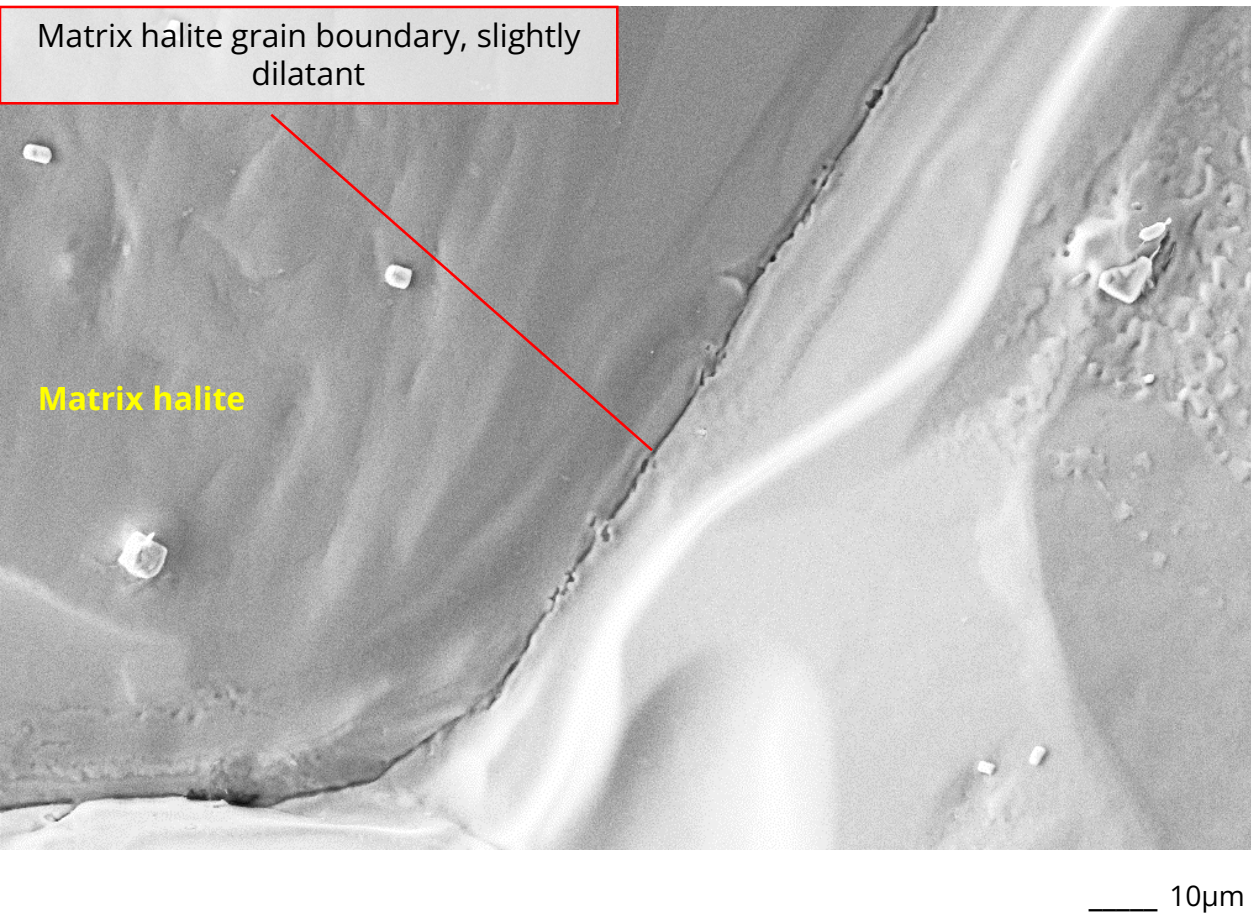
Grain boundary structure: **Zuidwending-1_TC5_MaP_43_0.6MPa**



Grain boundary structure: **Zuidwending-1_TC5_MaP_43_0.6MPa**



Grain boundary structure: **Zuidwending-1_TC5_MaP_43_0.6MPa**



Zuidwending-2_TC2_MaP_40_0.6MPa

Core piece photography: **Zuidwending-2_TC2_MaP_40_0.6MPa**

Reflected light



Transmitted light



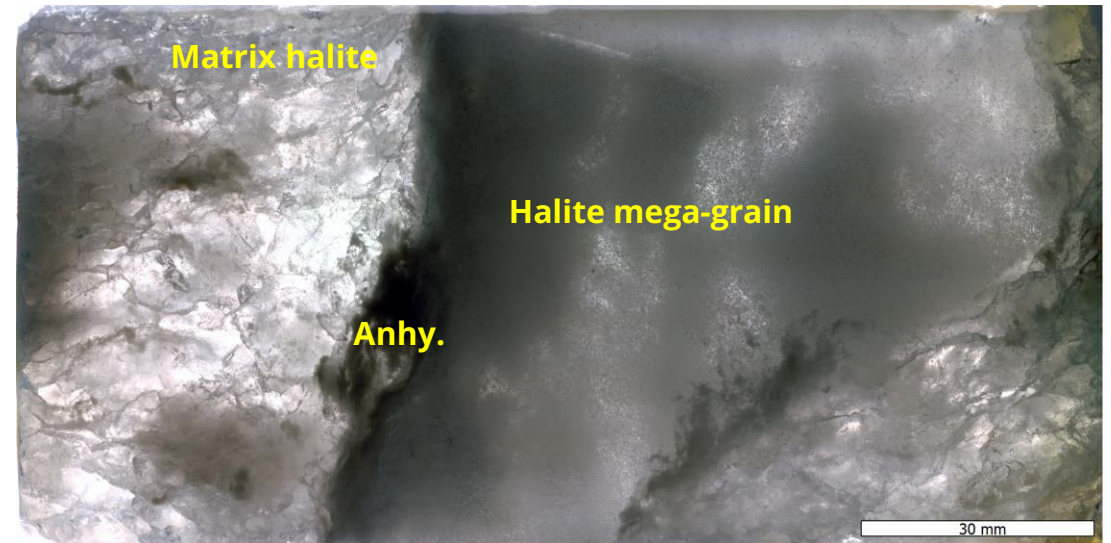
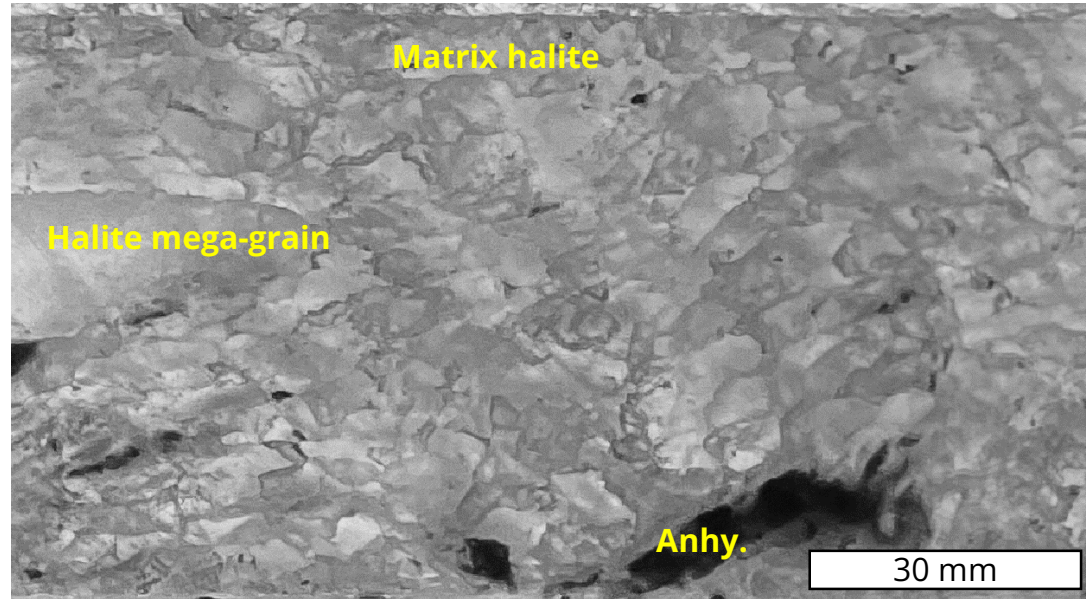
Transmitted light
(cut-side)



Reflected light
(cut-side)



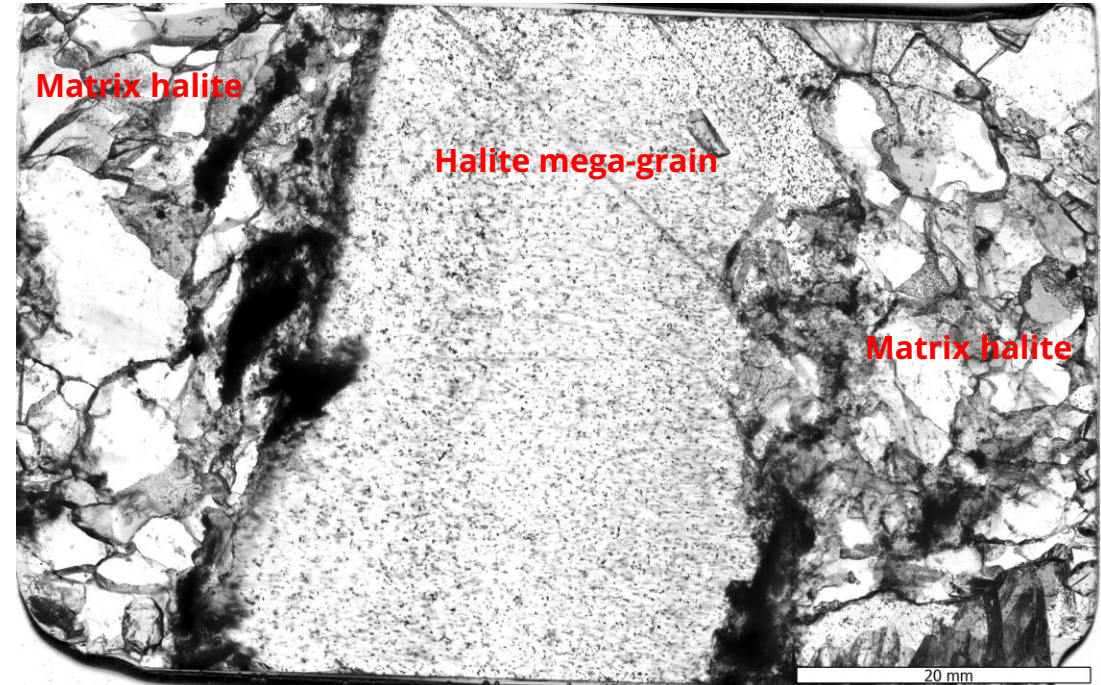
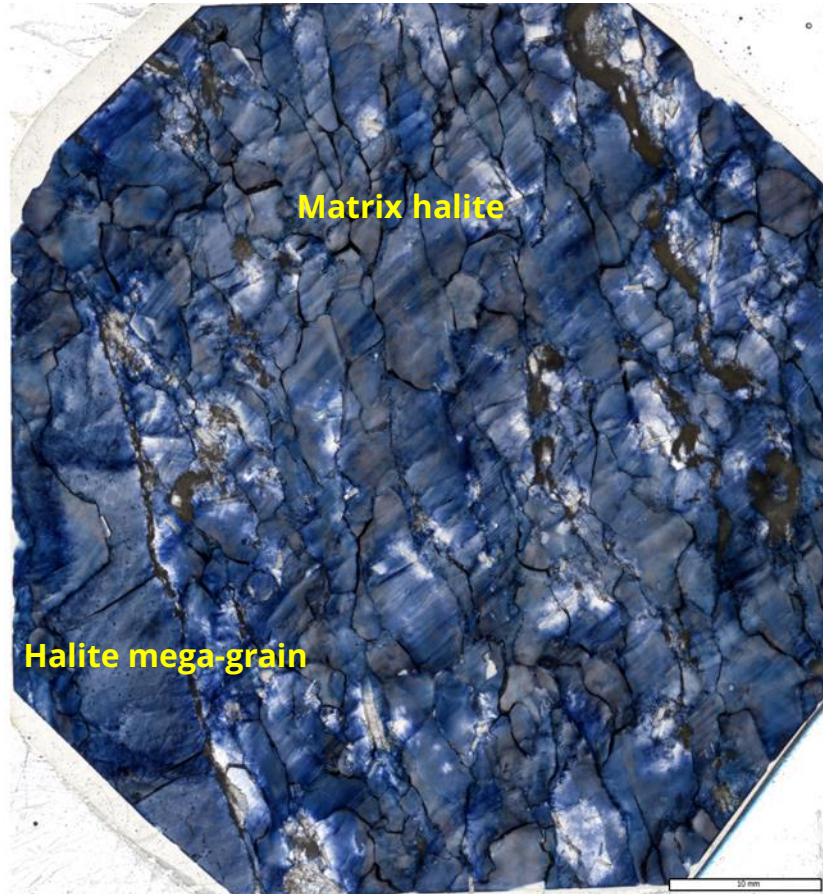
Core piece slab photo: Zuidwending-2_TC2_MaP_40_0.6MPa



Tectonically-deformed twin: ZW-MaP_05g

Lab-deformed twin: Zuidwending-2_TC2_MaP_40

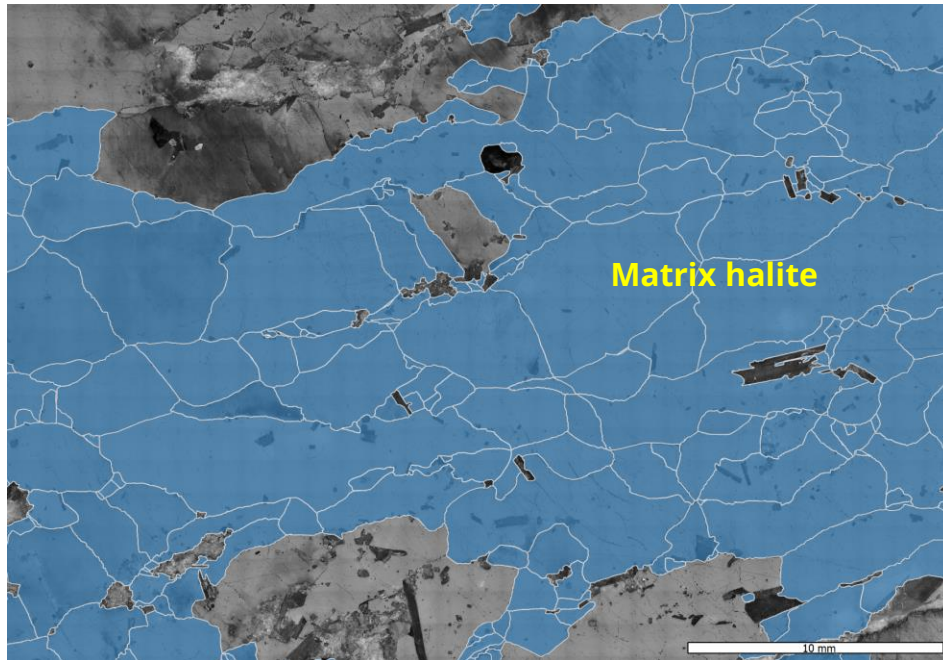
Thin section in transmitted light: **Zuidwending-2_TC2_MaP_40_0.6MPa**



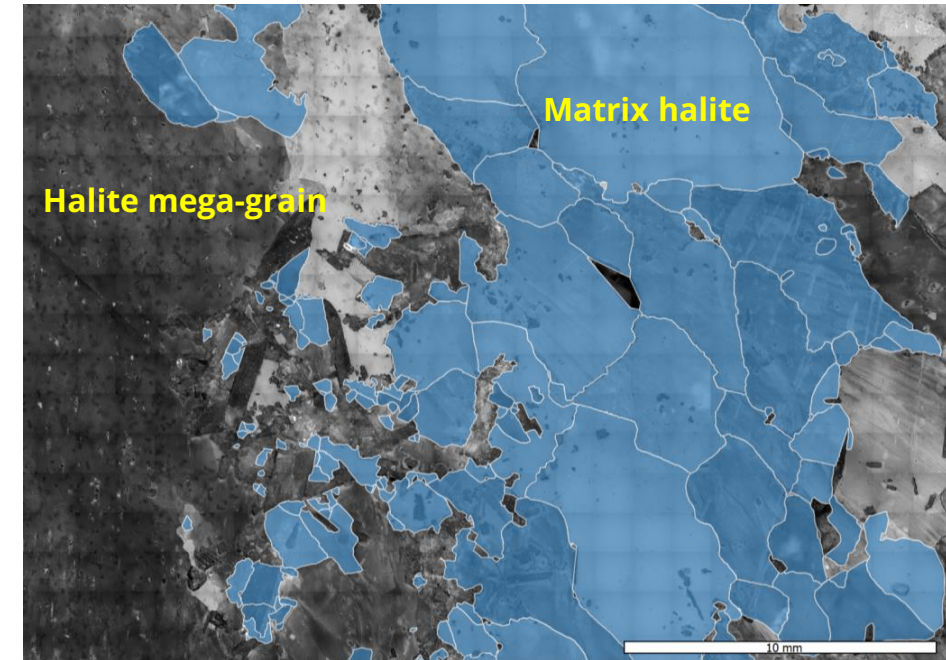
Tectonically-deformed twin: ZW-MaP_05g

Lab-deformed twin: Zuidwending-2_TC2_MaP_40

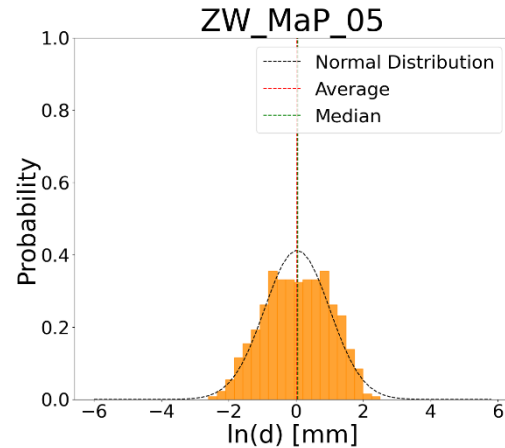
Grain size (d) distribution: Zuidwending-2_TC2_MaP_40_0.6MPa



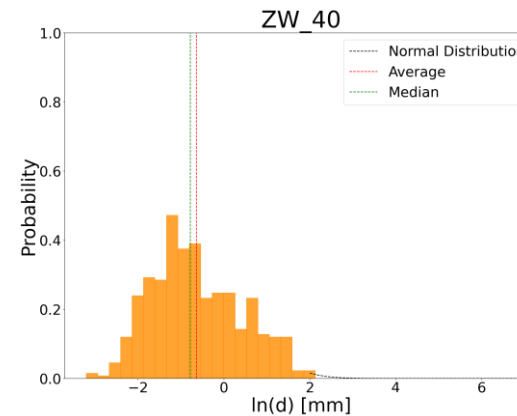
Tectonically-deformed twin: ZW-MaP_05g



Lab-deformed twin: Lab-deformed twin:
Zuidwending-2_TC2_MaP_40

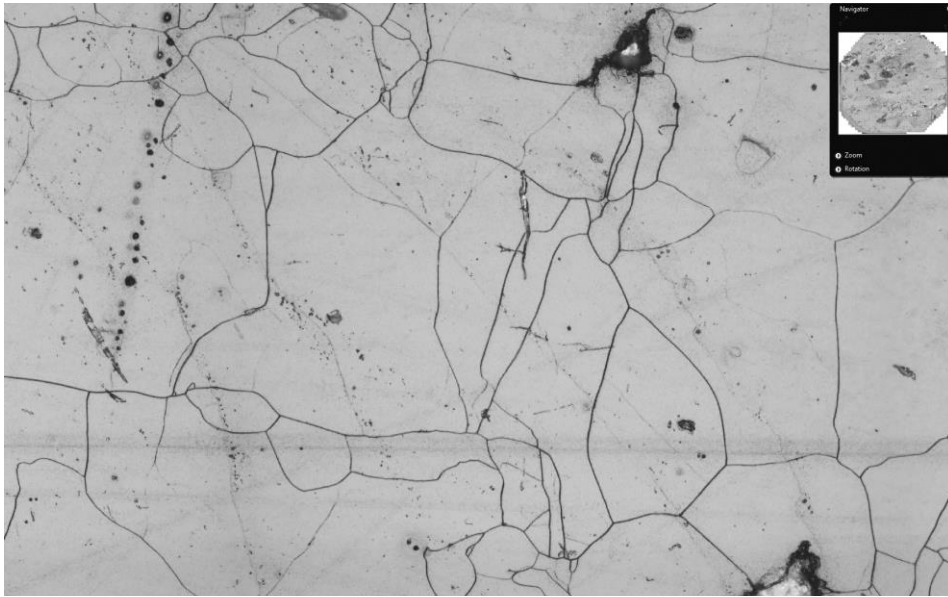


	d [mm]	ln(d)[mm]
Number of grains	507	507
Min [mm]	0.07	-2.61
Max [mm]	12.18	2.50
Median [mm]	1.05	0.05
Average [mm]	1.60	0.03
Std_deviation [mm]	1.54	0.97
Mode [mm]	1.51	0.41



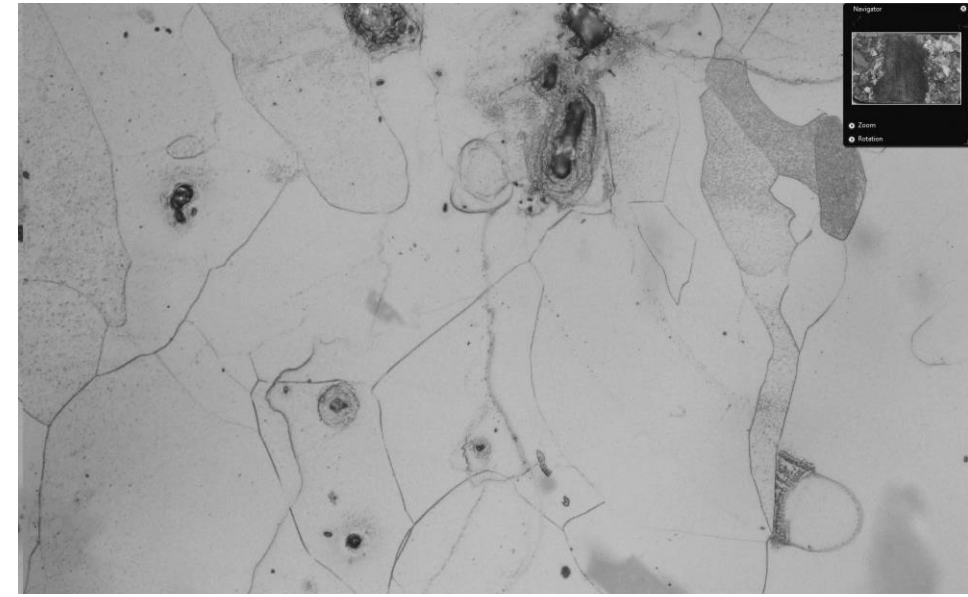
	d [mm]	ln(d)[mm]
Number of grains	501	501
Min [mm]	0.04	-3.20
Max [mm]	8.37	2.12
Median [mm]	0.46	-0.79
Average [mm]	0.94	-0.64
Std_deviation [mm]	1.19	1.05
Mode [mm]	0.84	-0.17

Subgrain size (D) distribution: **Zuidwending-2_TC2_MaP_40_0.6MPa**



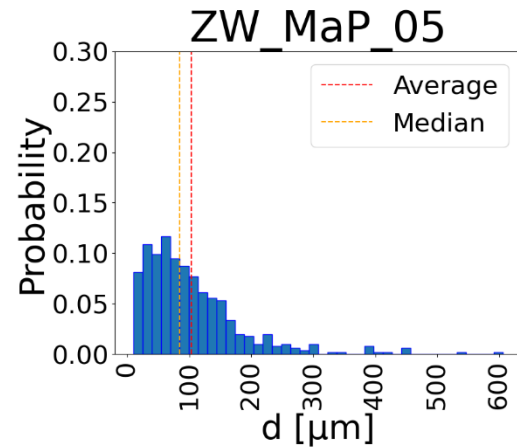
100µm

Tectonically-deformed twin: ZW-MaP_05g

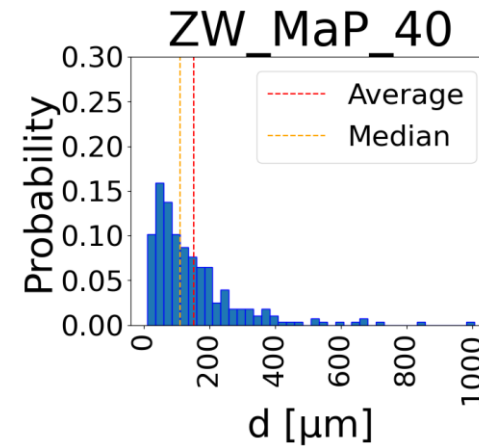


50µm

Lab-deformed twin: Zuidwending-2_TC2_MaP_40

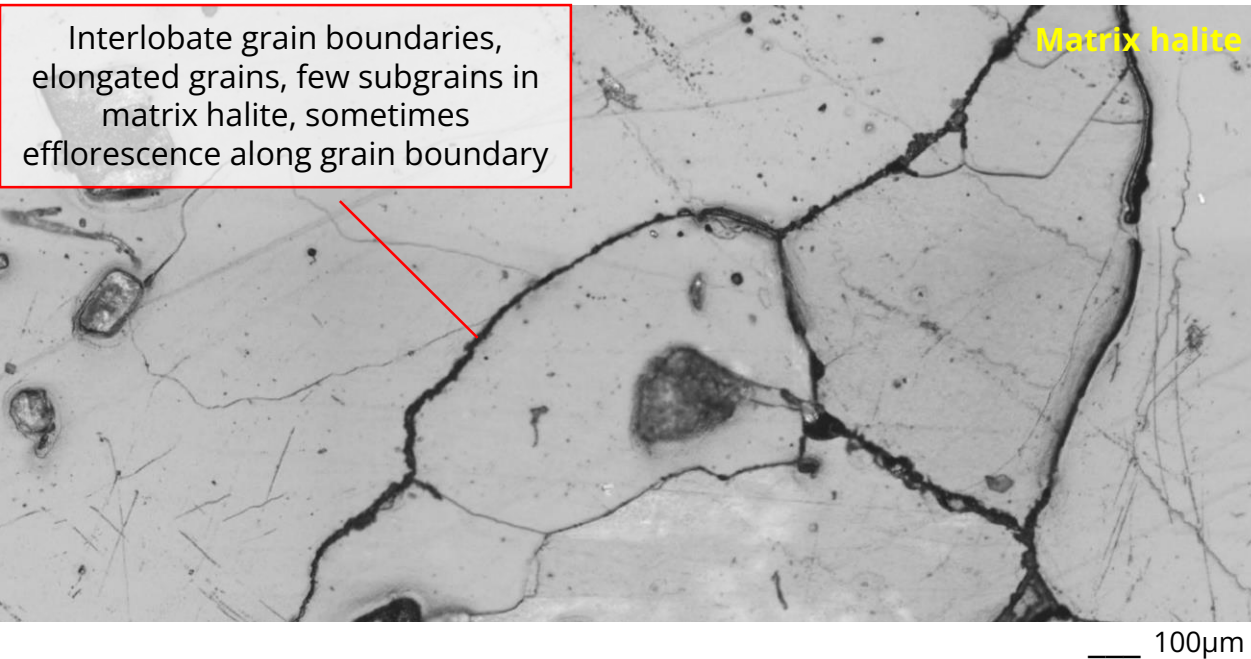


	d [µm]	ln(d)[µm]
Number of grains	505	505
Min [µm]	9.93	2.30
Max [µm]	605.78	6.41
Median [µm]	84.39	4.44
Average [µm]	103.75	4.37
Std_deviation [µm]	81.55	0.77
Mode [µm]	64.63	4.17

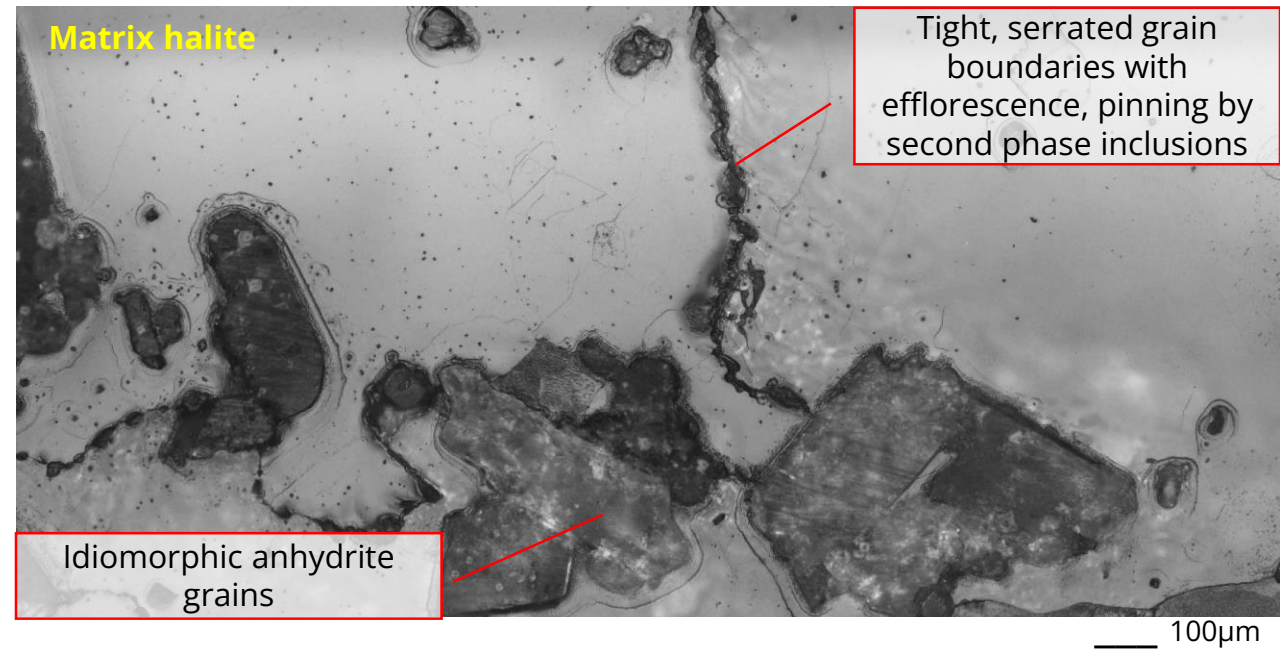


	d [µm]	ln(d)[µm]
Number of grains	303	303
Min [µm]	10.56	2.36
Max [µm]	1,005.04	6.91
Median [µm]	103.17	4.64
Average [µm]	144.58	4.61
Std_deviation [µm]	138.04	0.87
Mode [µm]	92.43	4.53

Texture: Zuidwending-2_TC2_MaP_40_0.6MPa

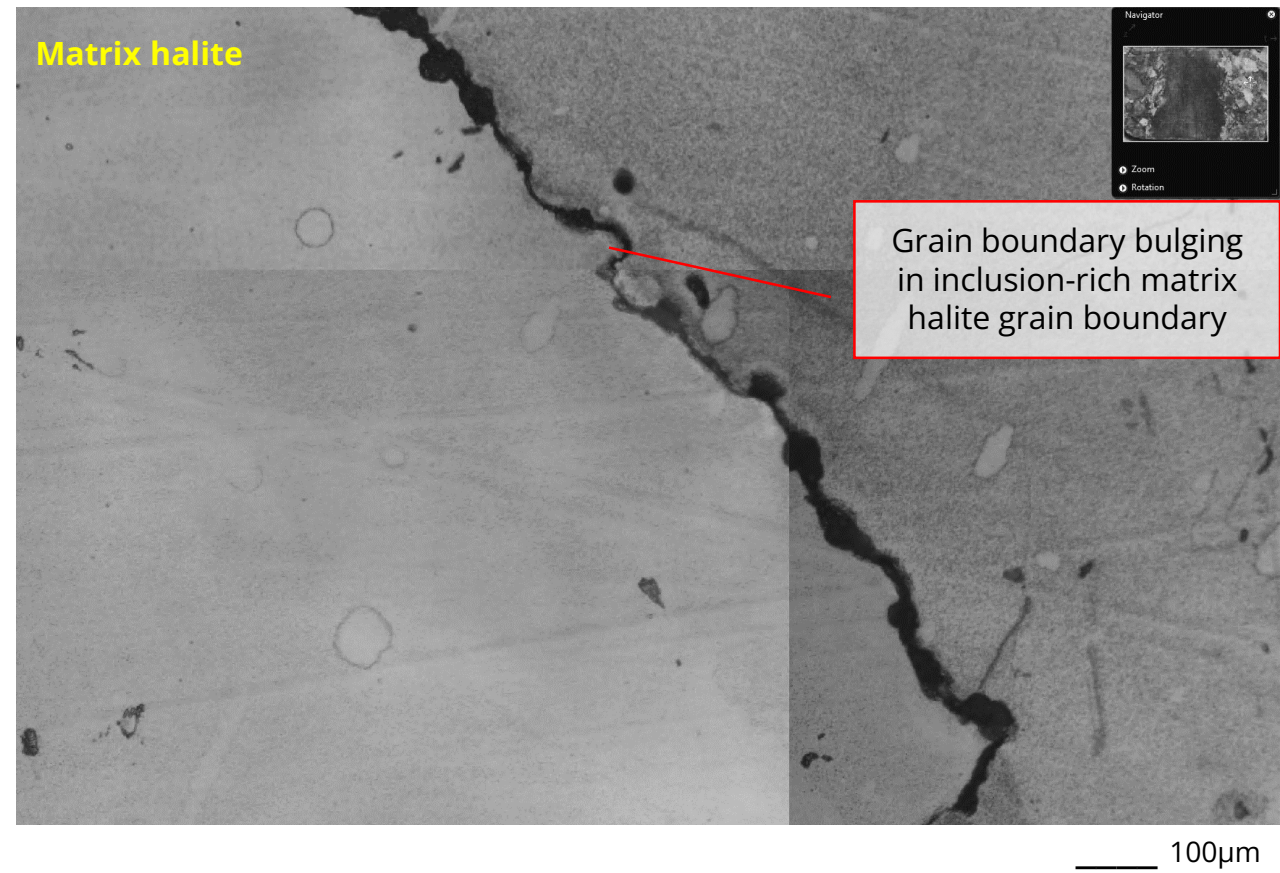
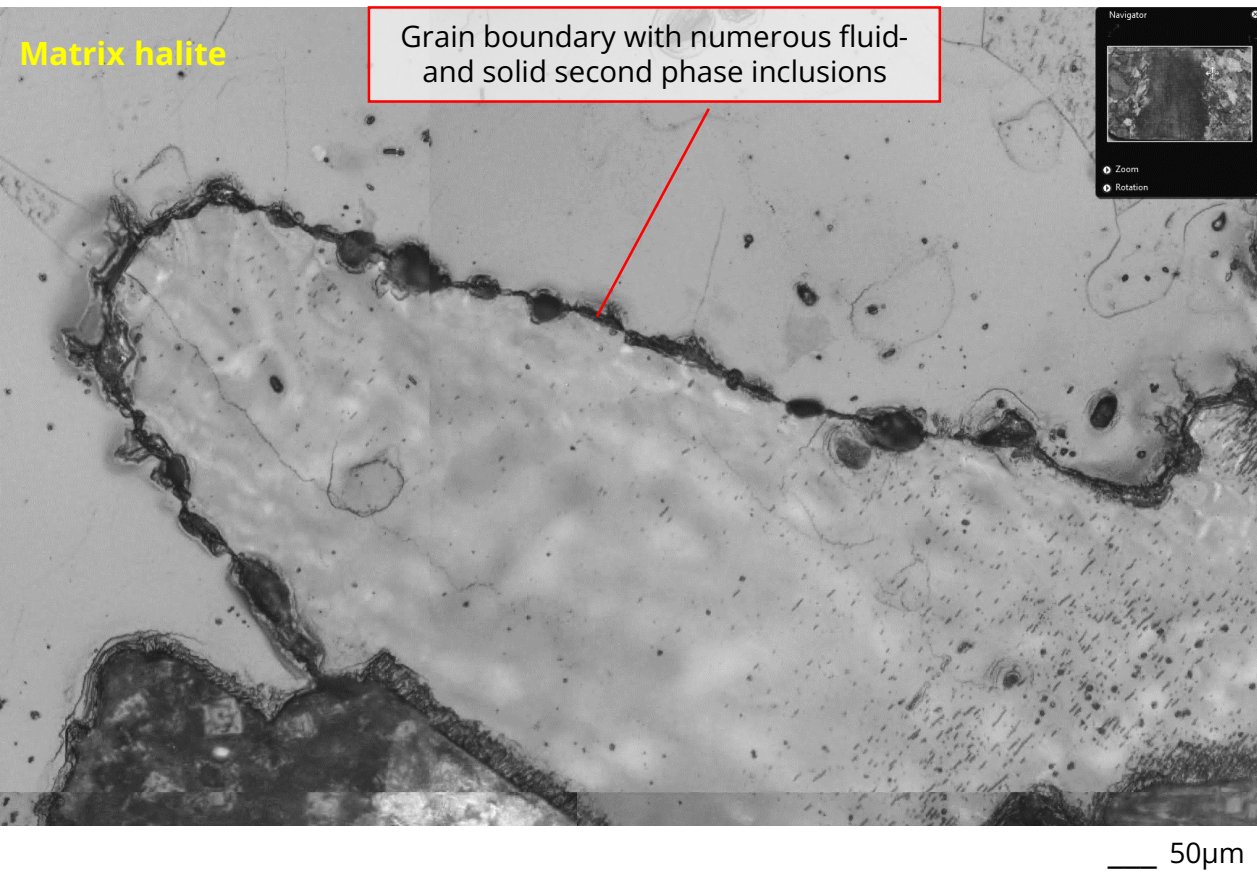


Tectonically-deformed twin: ZW-MaP_05g

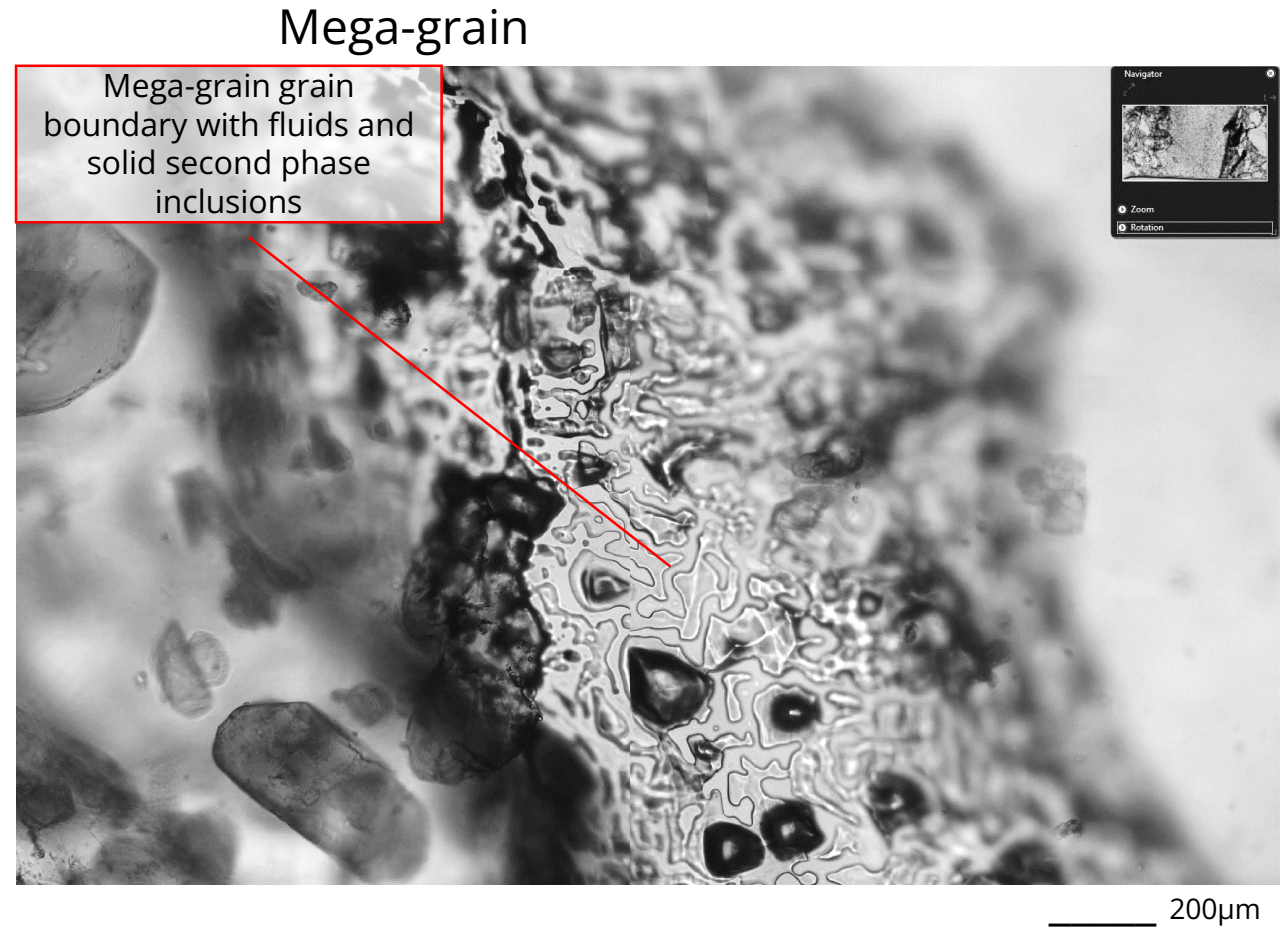
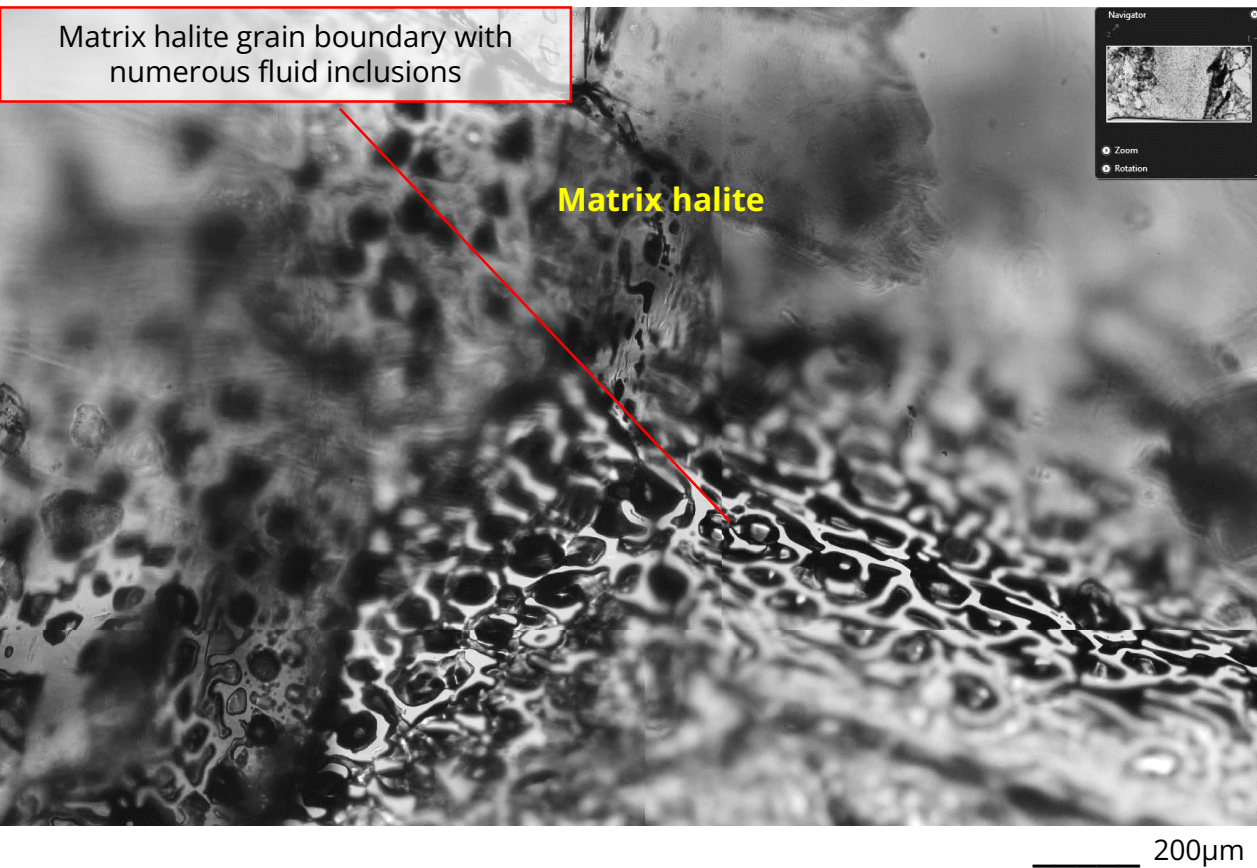


Lab-deformed twin: Lab-deformed twin: Zuidwending-2_TC2_MaP_40

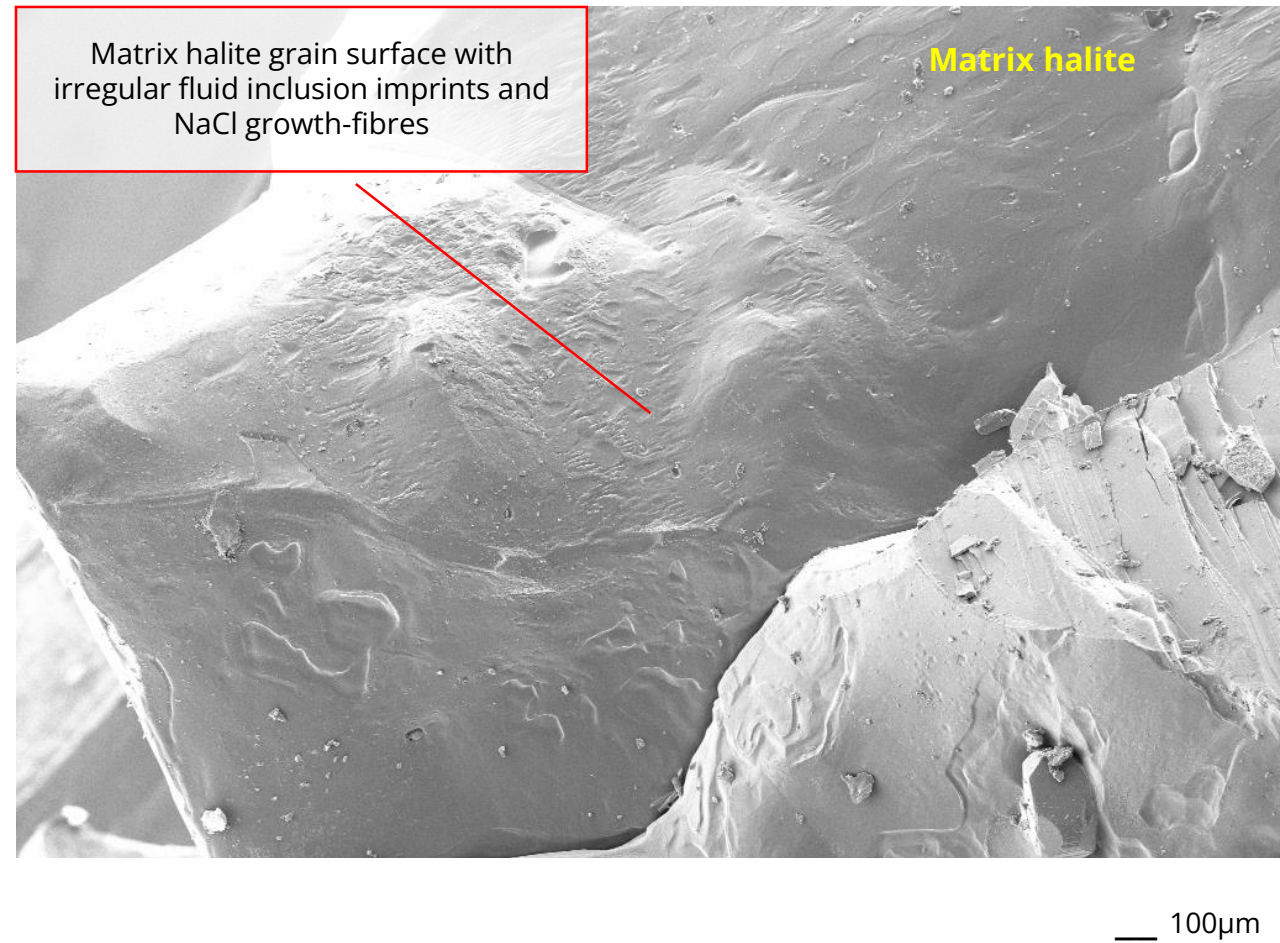
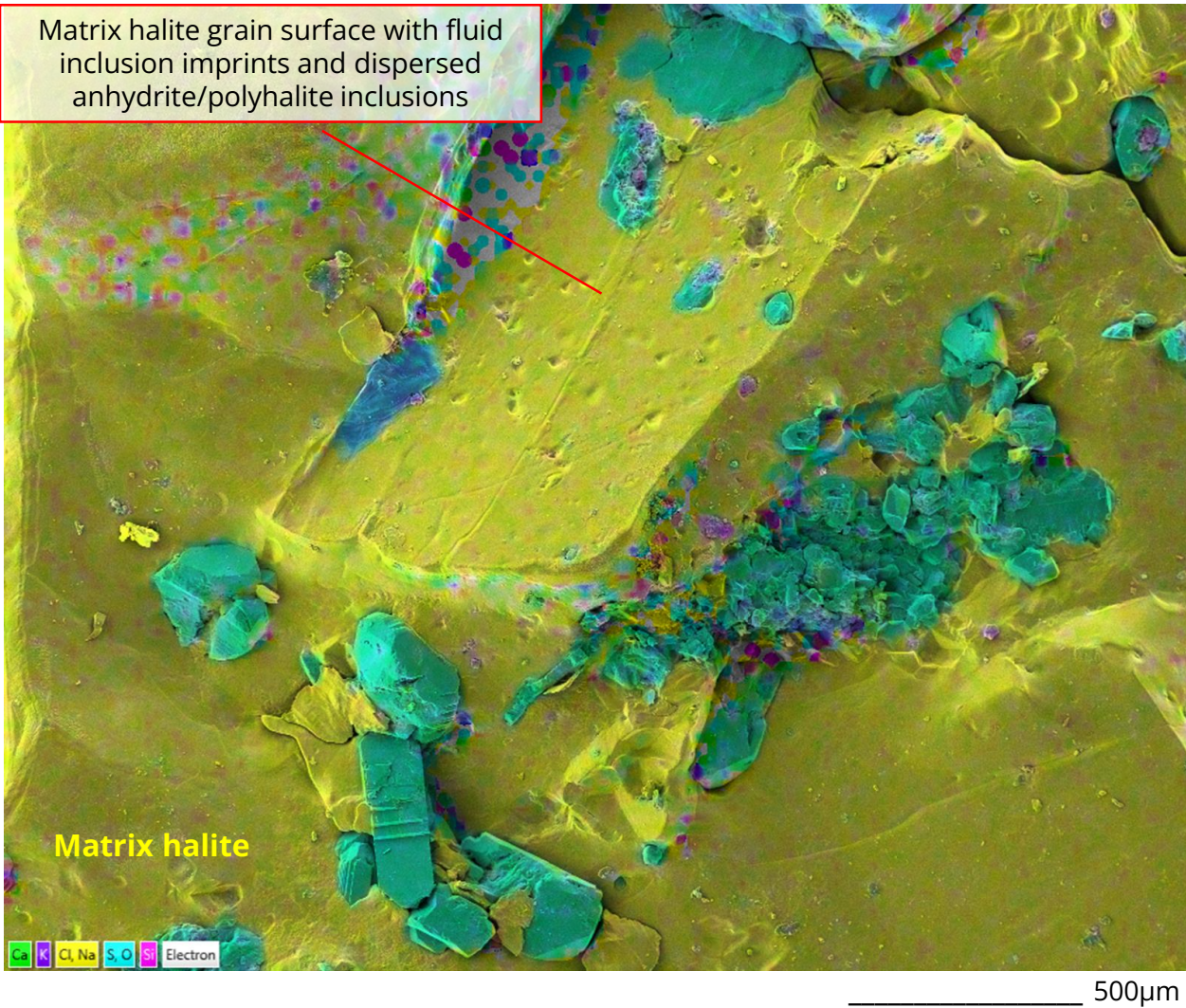
Grain boundary structure: Zuidwending-2_TC2_MaP_40_0.6MPa



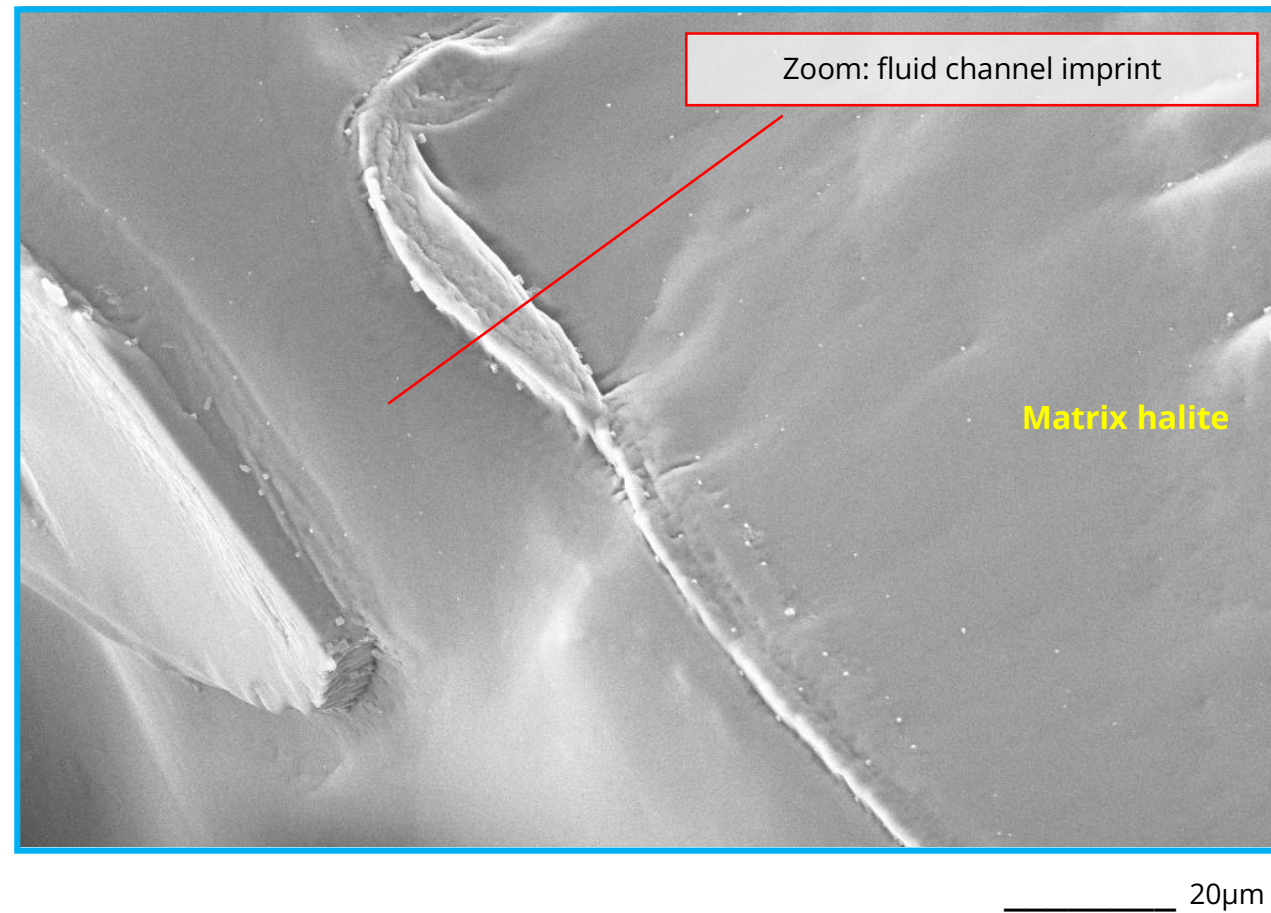
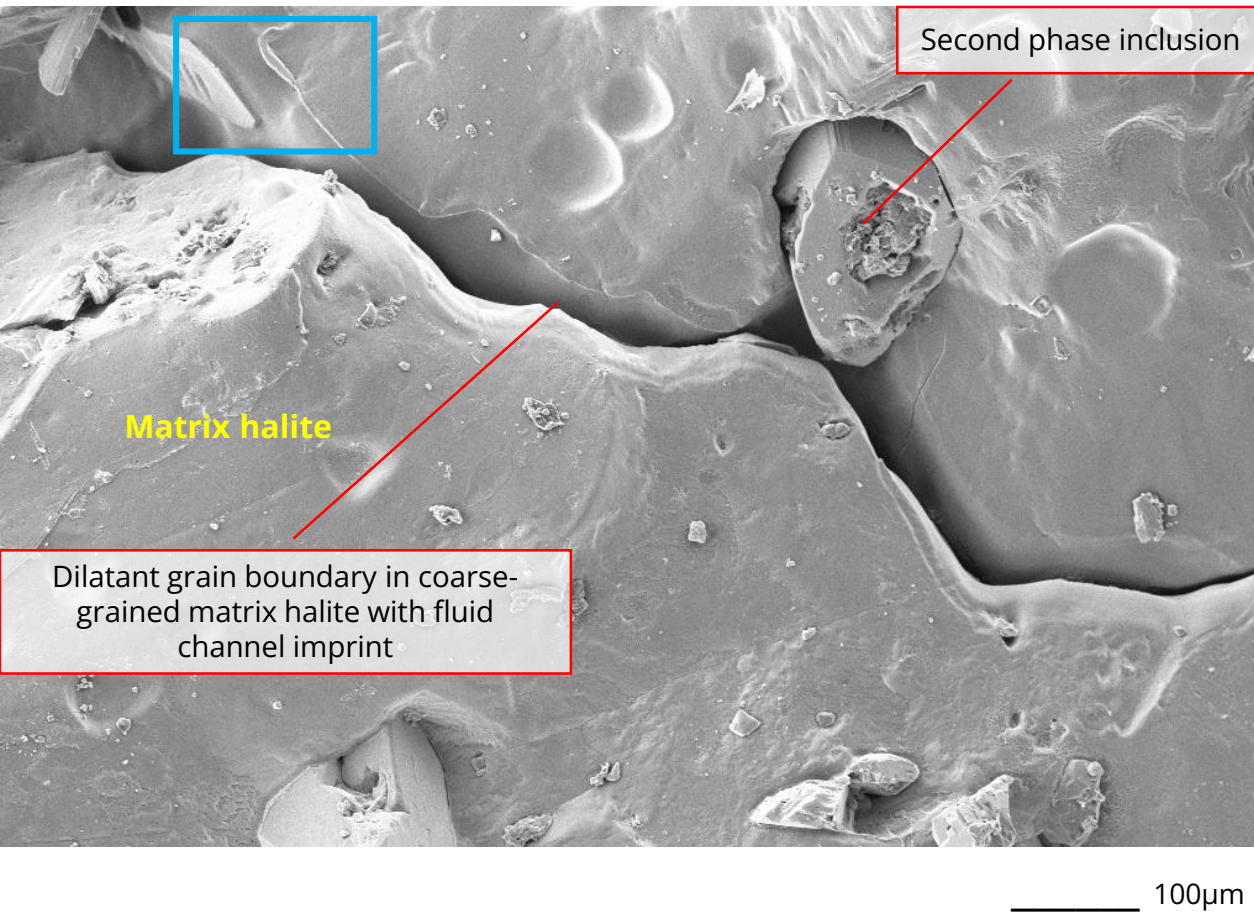
Grain boundary structure: **Zuidwending-2_TC2_MaP_40_0.6MPa**



Grain boundary structure: **Zuidwending-2_TC2_MaP_40_0.6MPa**



Grain boundary structure: **Zuidwending-2_TC2_MaP_40_0.6MPa**



Zuidwending-3_TC4_MaP_42_3.0MPa

Core piece photography: **Zuidwending-3_TC4_MaP_42_3.0MPa**

Reflected light



Transmitted light



Transmitted light
(cut-side)



Reflected light
(cut-side)



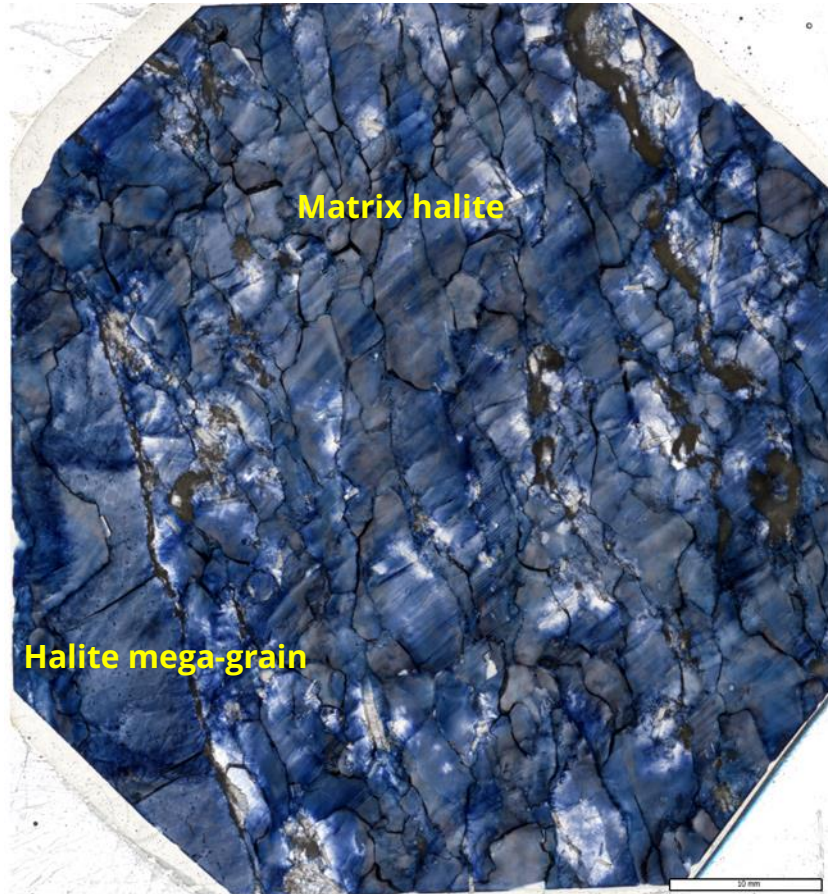
Core piece slab photo: **Zuidwending-3_TC4_MaP_42_3.0MPa**



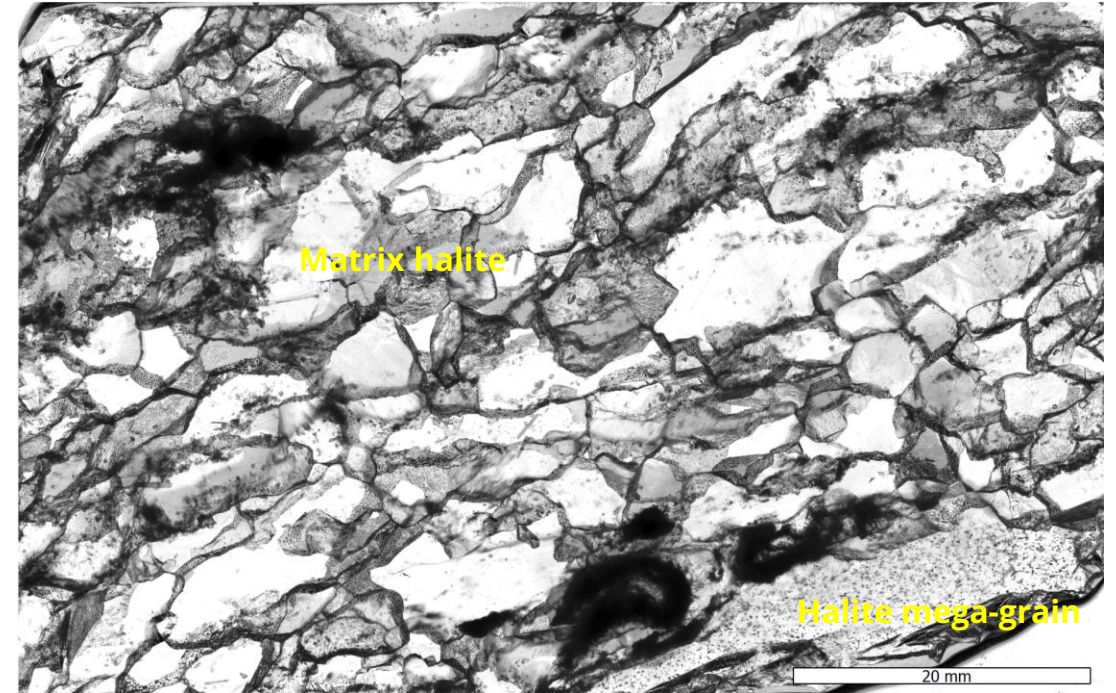
Tectonically-deformed twin: ZW-MaP_05g

Lab-deformed twin: Zuidwending-3_TC4_MaP_42

Thin section in transmitted light: **Zuidwending-3_TC4_MaP_42_3.0MPa**

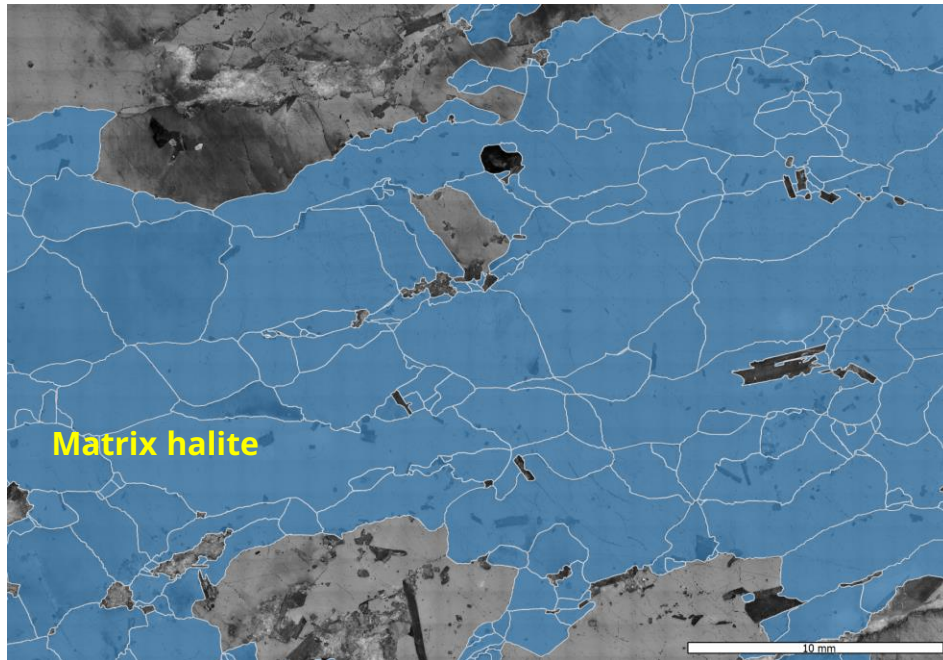


Tectonically-deformed twin: ZW-MaP_05g

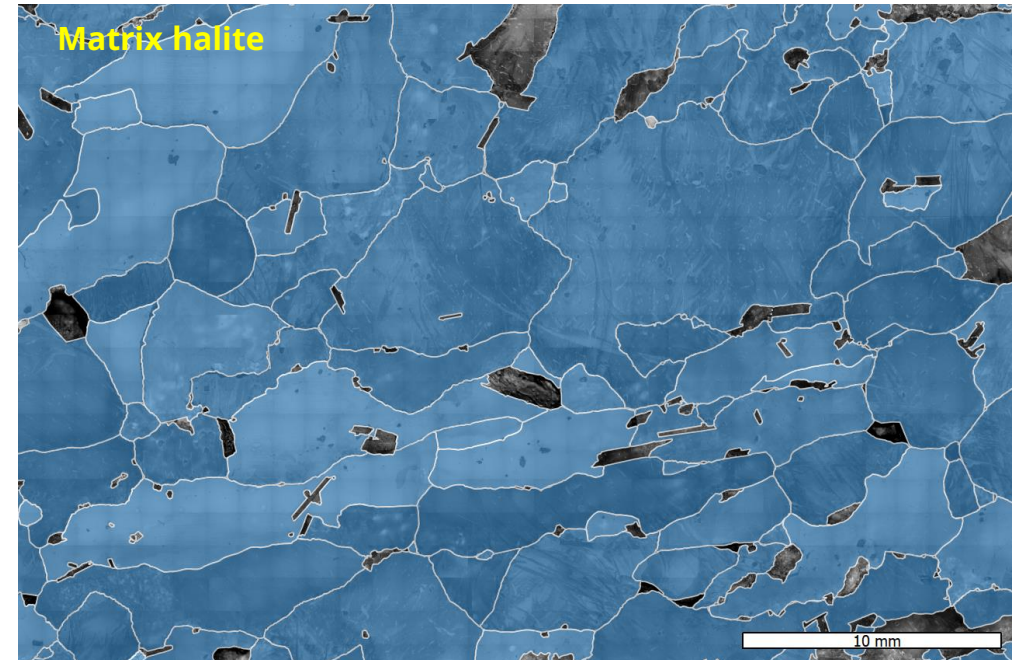


Lab-deformed twin: Zuidwending-3_TC4_MaP_42

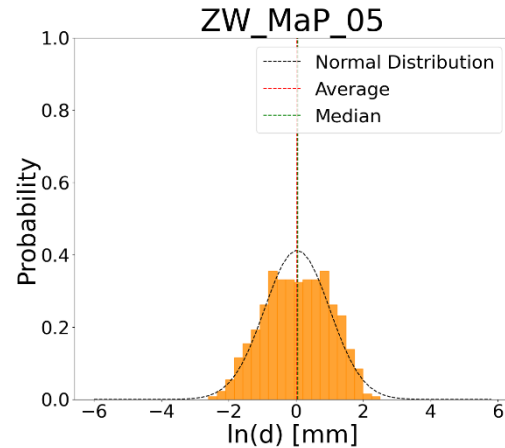
Grain size (d) distribution: Zuidwending-3_TC4_MaP_42_3.0MPa



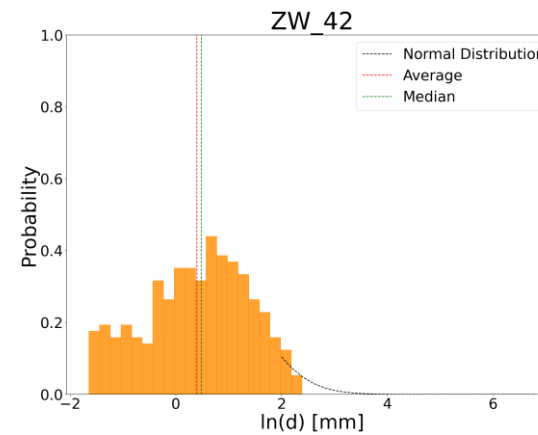
Tectonically-deformed twin: ZW-MaP_05g



Lab-deformed twin: Lab-deformed twin:
Zuidwending-3_TC4_MaP_42

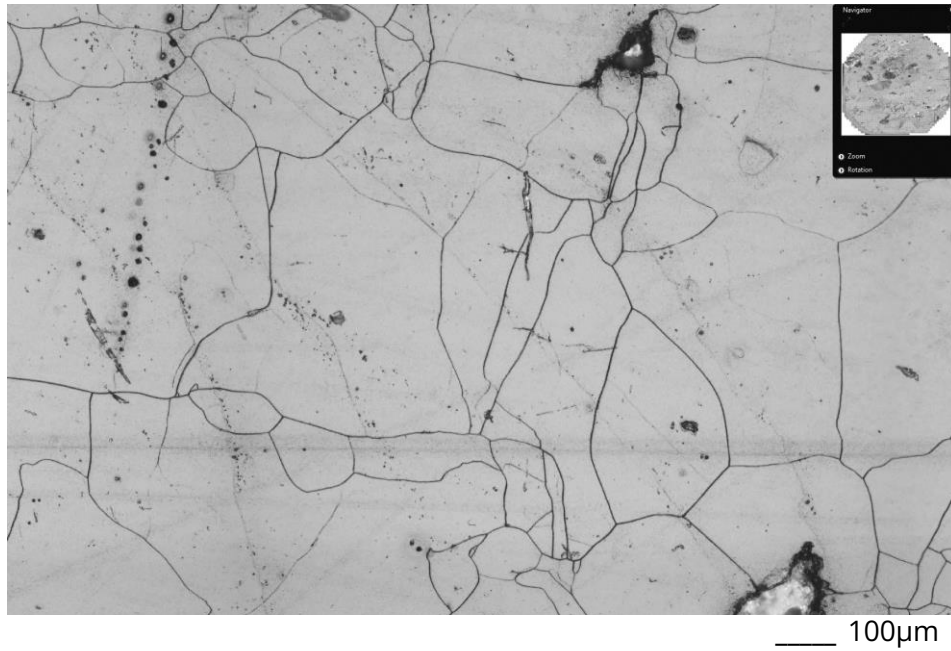


	d [mm]	ln(d)[mm]
Number of grains	507	507
Min [mm]	0.07	-2.61
Max [mm]	12.18	2.50
Median [mm]	1.05	0.05
Average [mm]	1.60	0.03
Std_deviation [mm]	1.54	0.97
Mode [mm]	1.51	0.41

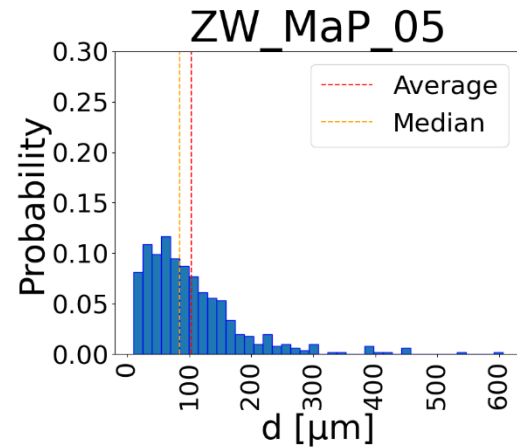


	d [mm]	ln(d)[mm]
Number of grains	502	502
Min [mm]	0.03	-3.50
Max [mm]	10.86	2.39
Median [mm]	0.61	-0.50
Average [mm]	1.41	-0.35
Std_deviation [mm]	1.78	1.19
Mode [mm]	2.01	0.70

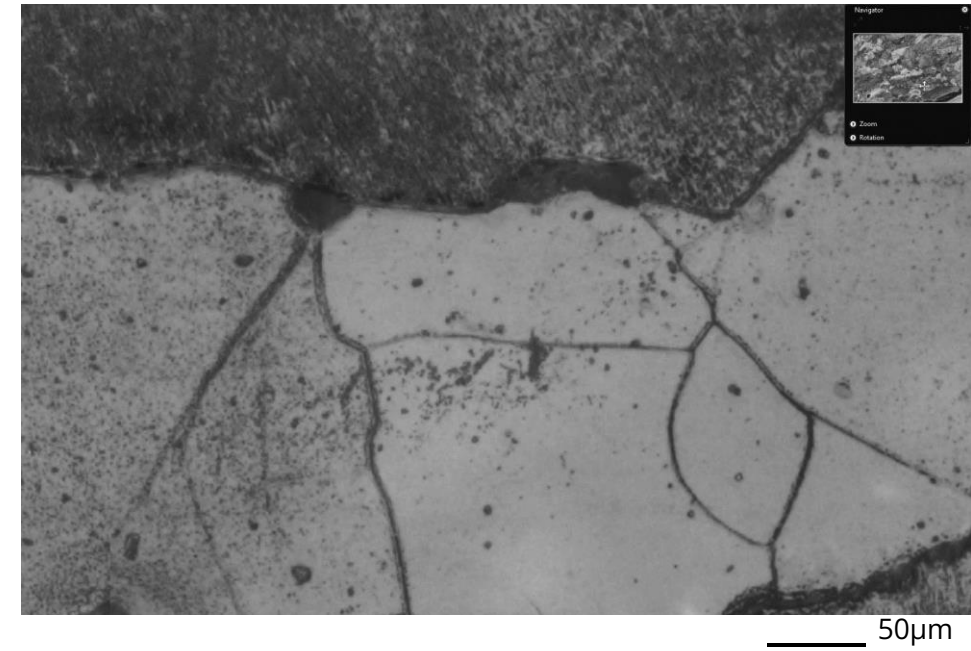
Subgrain size (D) distribution: **Zuidwending-3_TC4_MaP_42_3.0MPa**



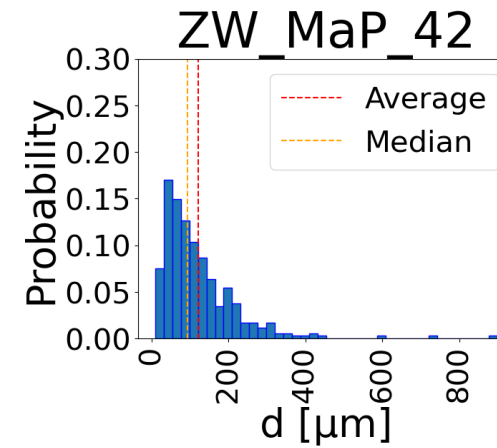
Tectonically-deformed twin: ZW-MaP_05g



	d [µm]	ln(d)[µm]
Number of grains	505	505
Min [µm]	9.93	2.30
Max [µm]	605.78	6.41
Median [µm]	84.39	4.44
Average [µm]	103.75	4.37
Std_deviation [µm]	81.55	0.77
Mode [µm]	64.63	4.17

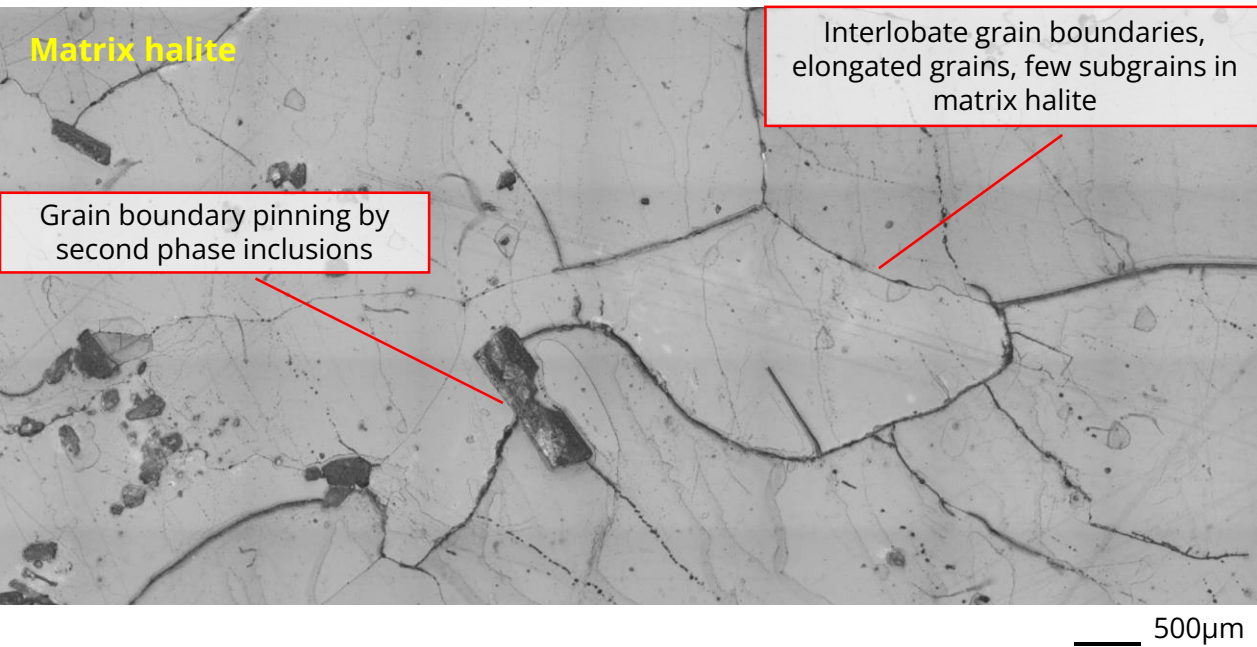


Lab-deformed twin: Zuidwending-3_TC4_MaP_42

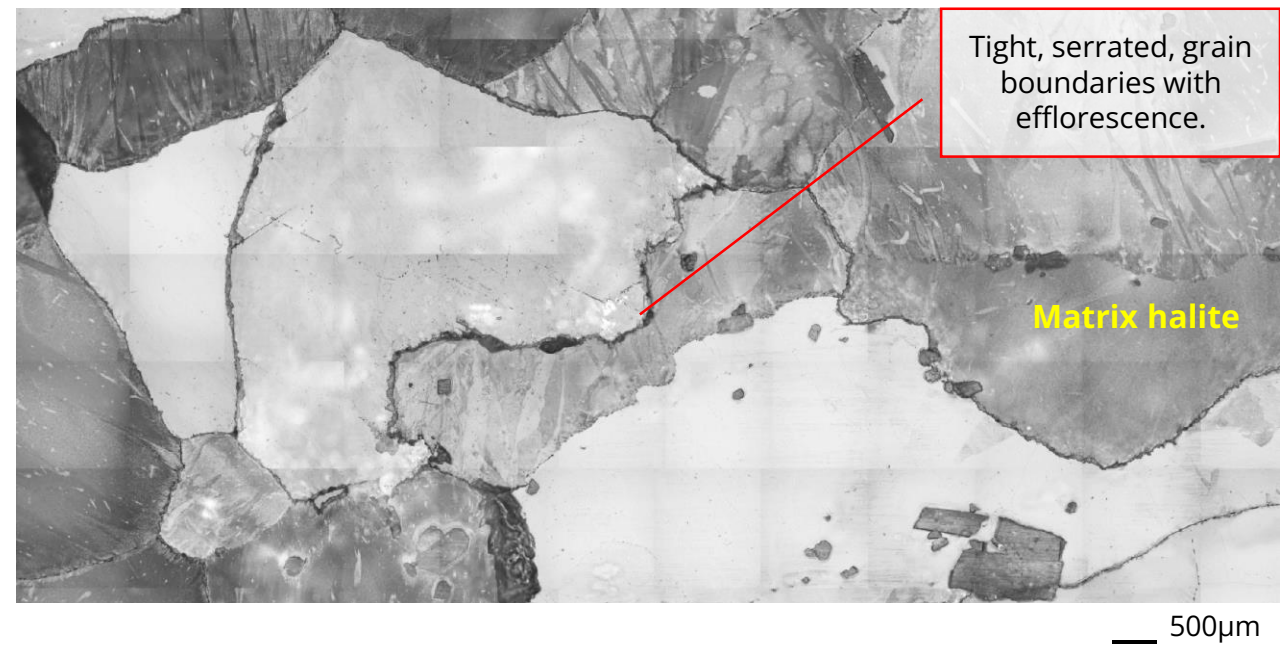


	d [µm]	ln(d)[µm]
Number of grains	427	427
Min [µm]	9.34	2.23
Max [µm]	898.49	6.80
Median [µm]	94.64	4.55
Average [µm]	132.04	4.57
Std_deviation [µm]	119.69	0.80
Mode [µm]	176.62	5.17

Texture: Zuidwending-3_TC4_MaP_42_3.0MPa

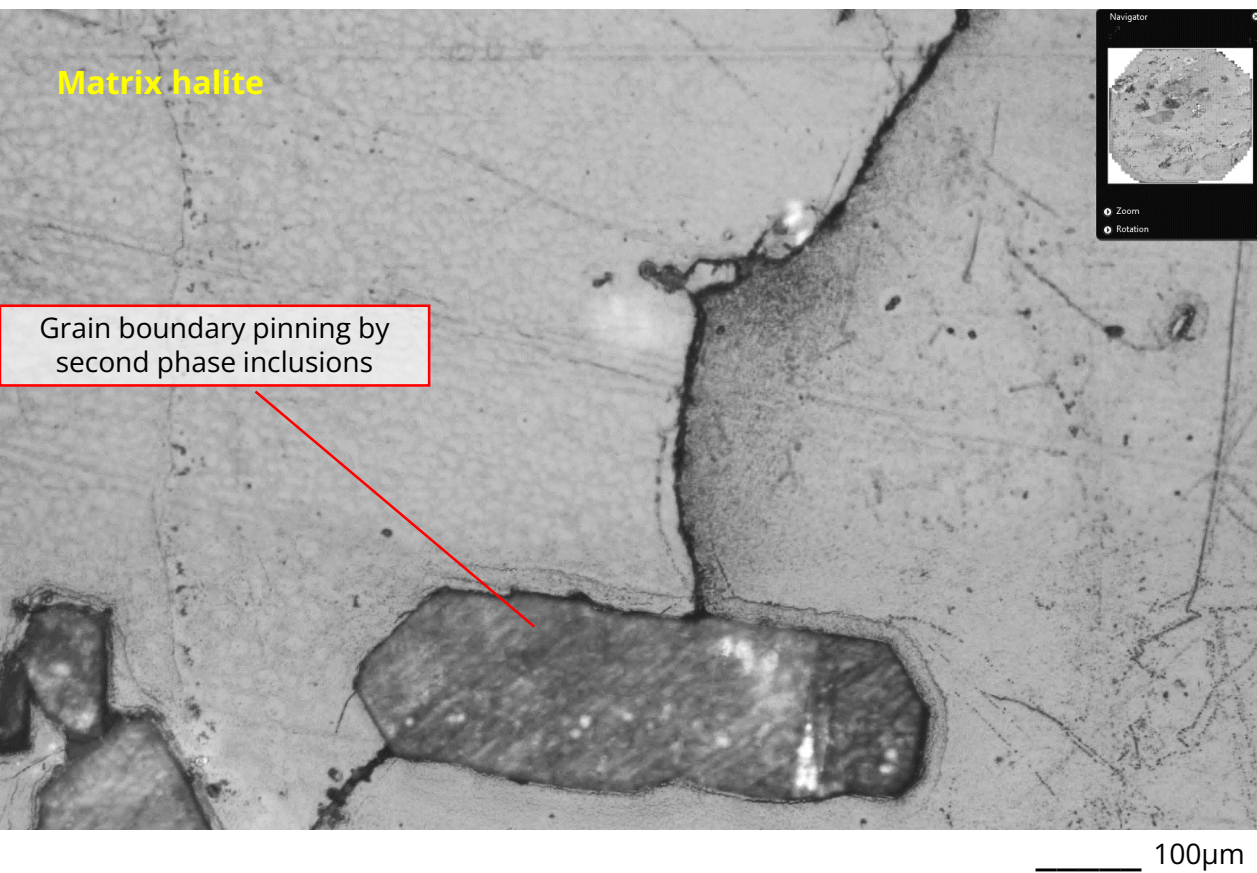


Tectonically-deformed twin: ZW-MaP_05g

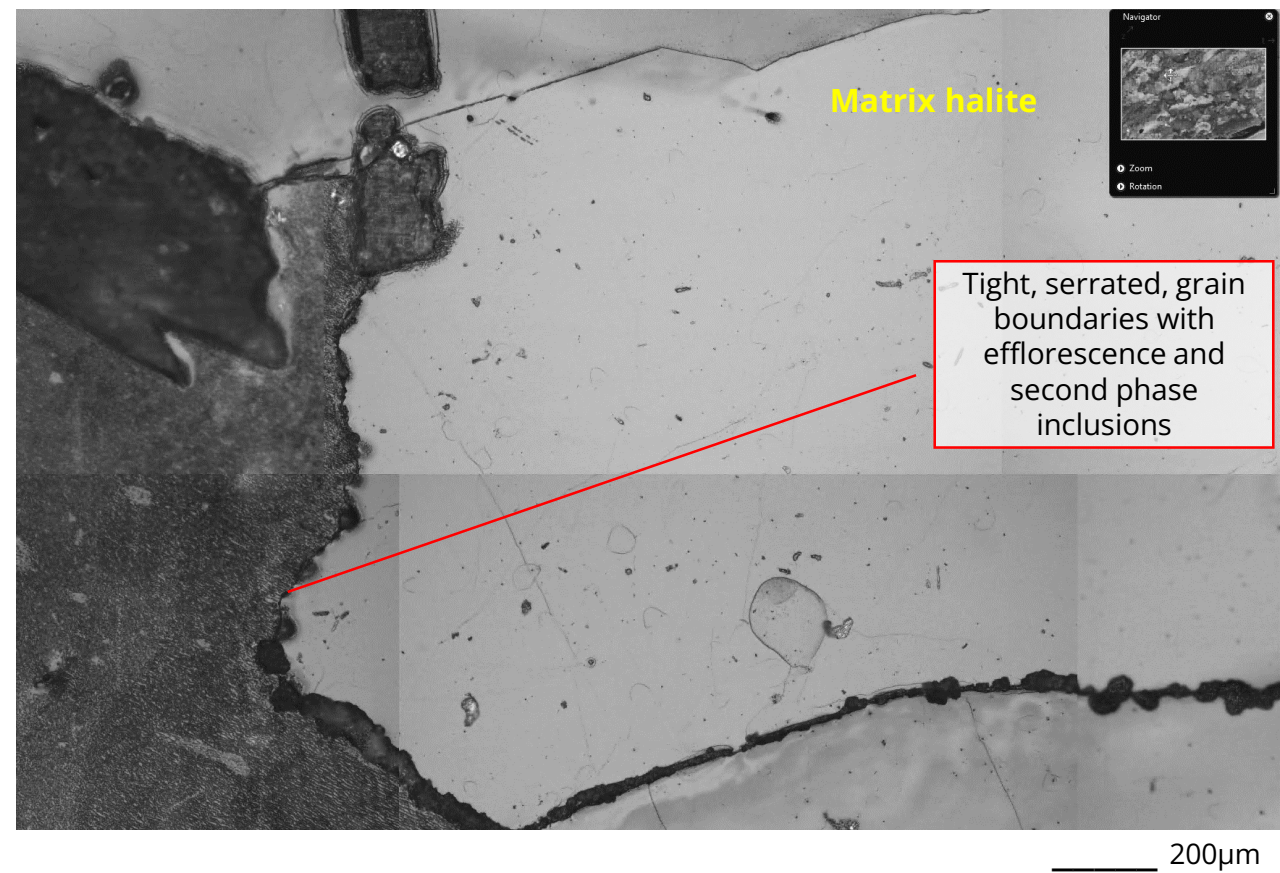


Lab-deformed twin: Zuidwending-3_TC4_MaP_42

Texture: Zuidwending-3_TC4_MaP_42_3.0MPa

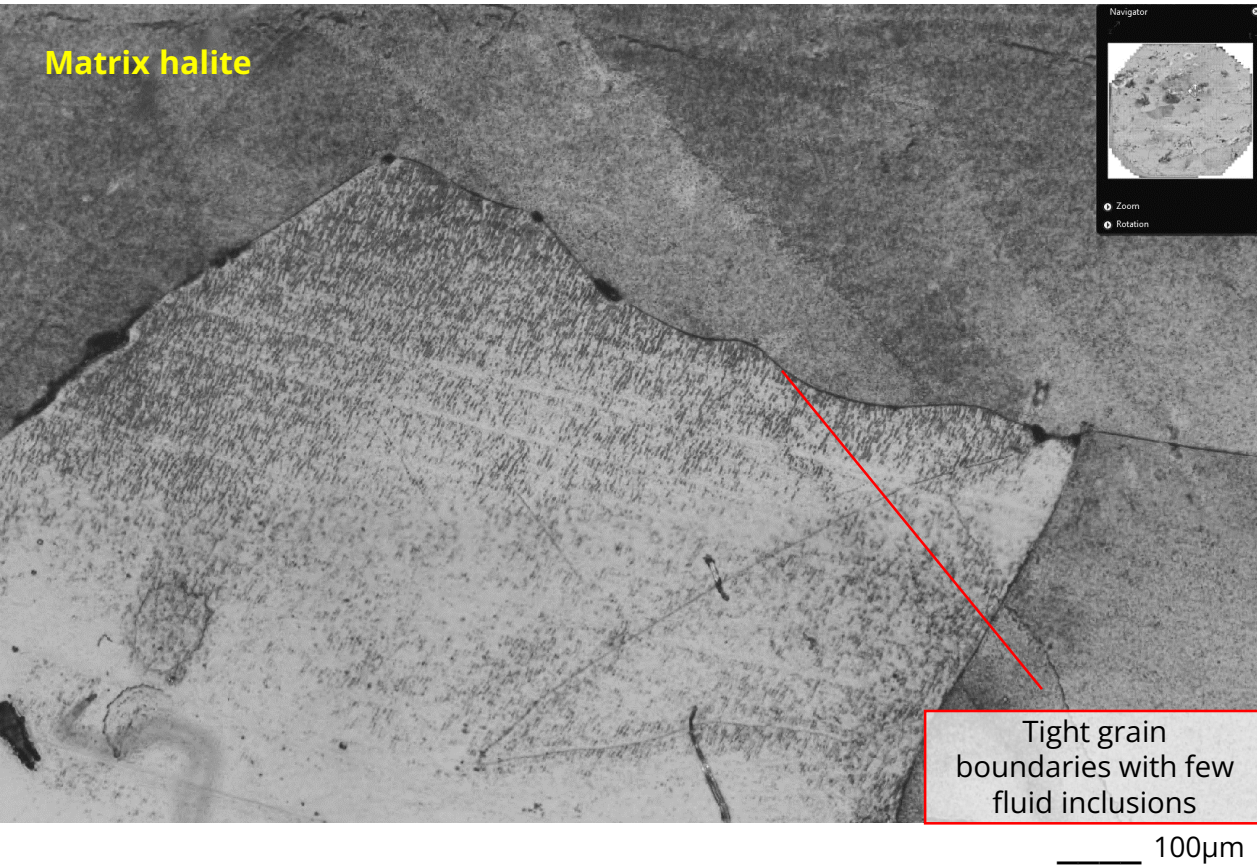


Tectonically-deformed twin: ZW-MaP_05g

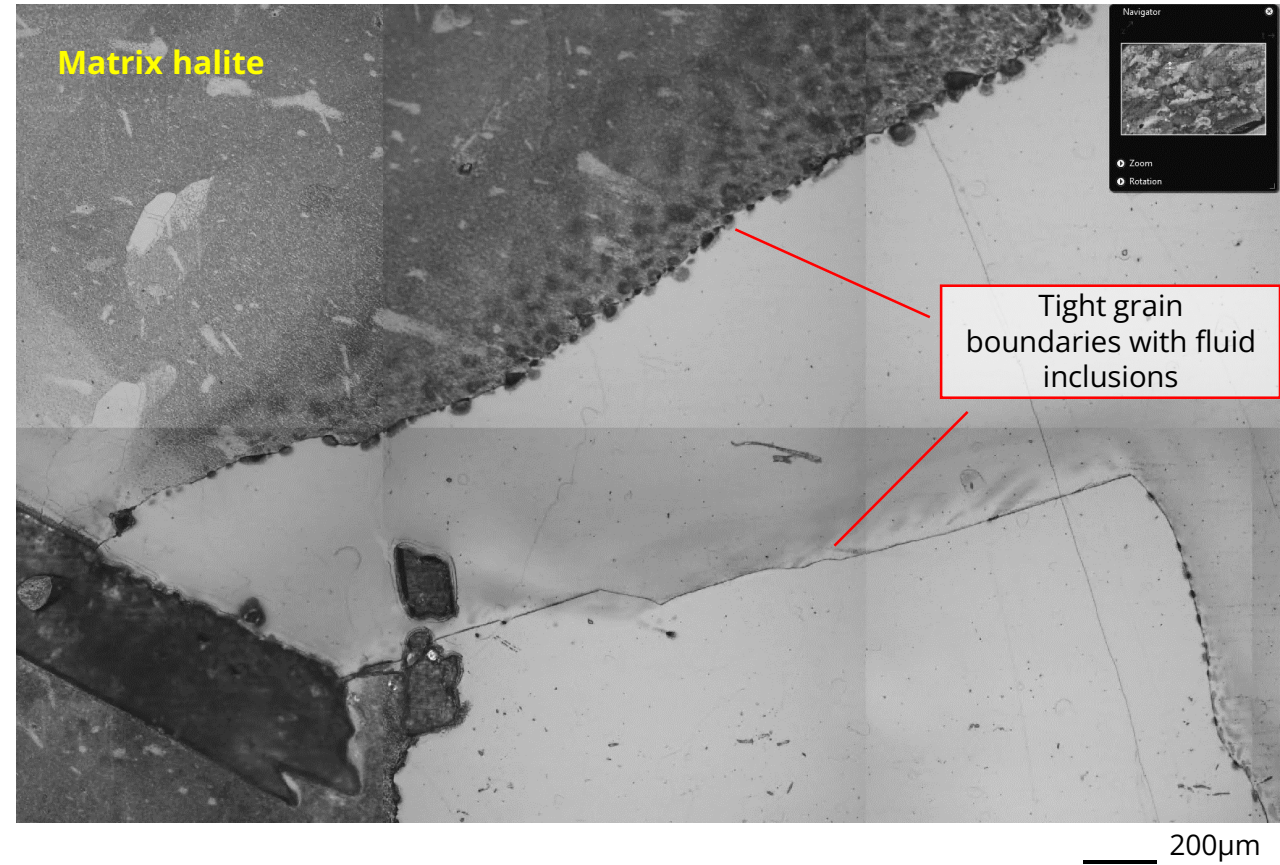


Lab-deformed twin: Zuidwending-3_TC4_MaP_42

Grain boundary structure: **Zuidwending-3_TC4_MaP_42_3.0MPa**

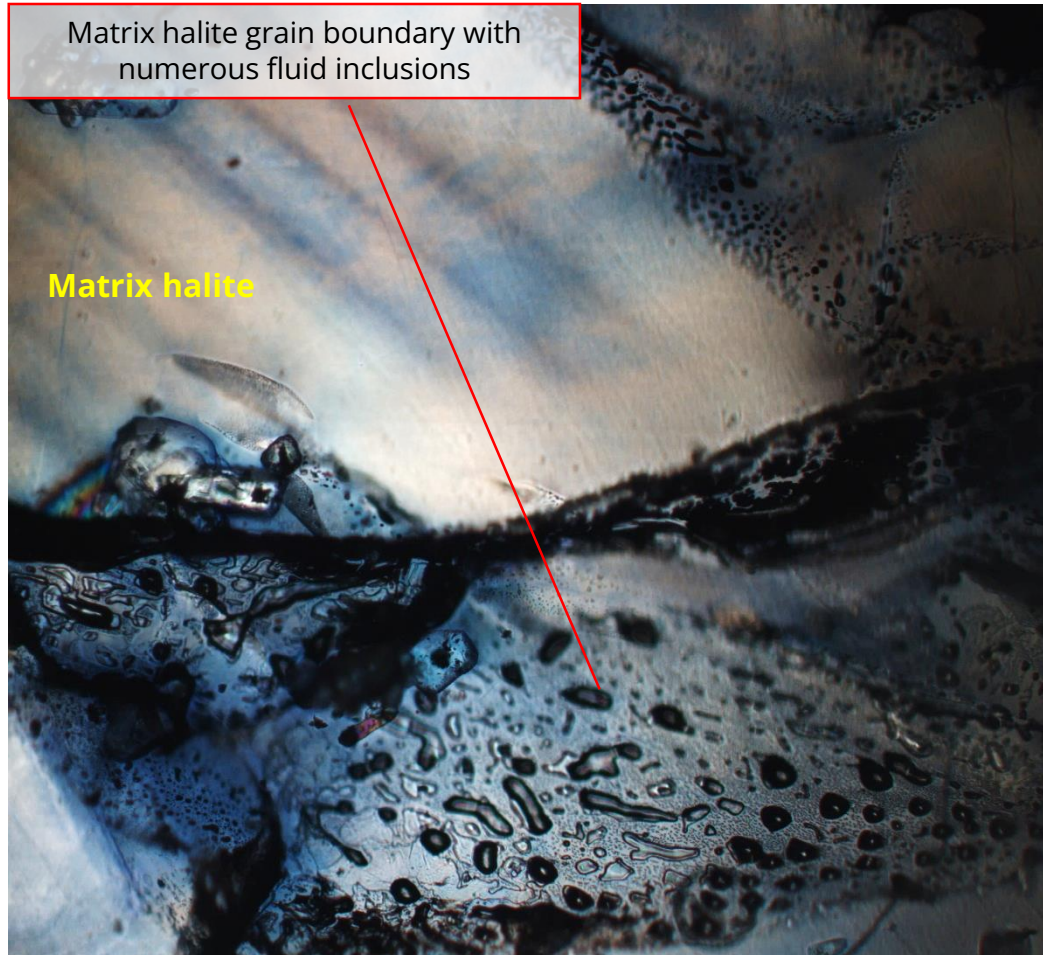


Tectonically-deformed twin: ZW-MaP_05g



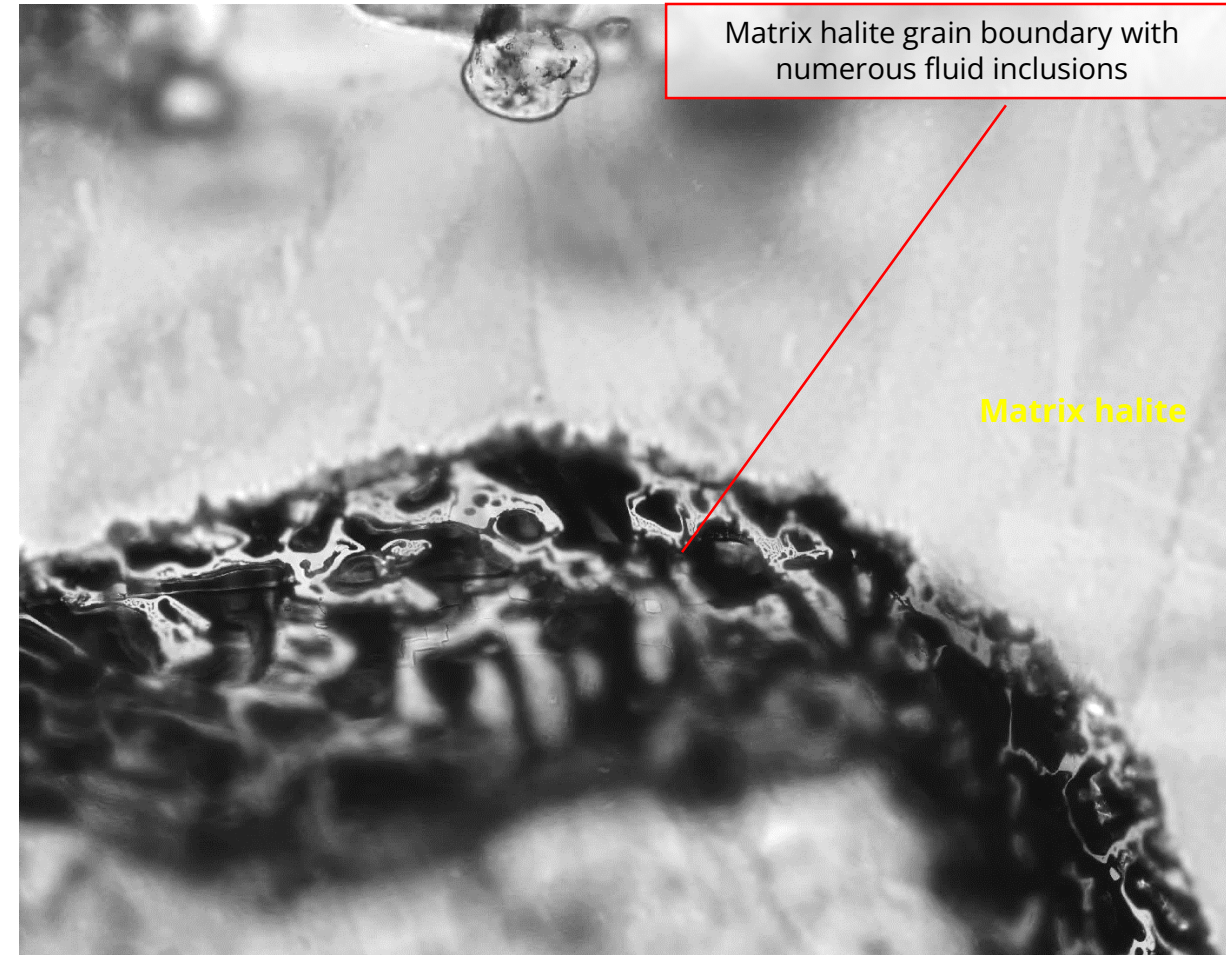
Lab-deformed twin: Zuidwending-3_TC4_MaP_42

Grain boundary structure: Zuidwending-3_TC4_MaP_42_3.0MPa



200µm

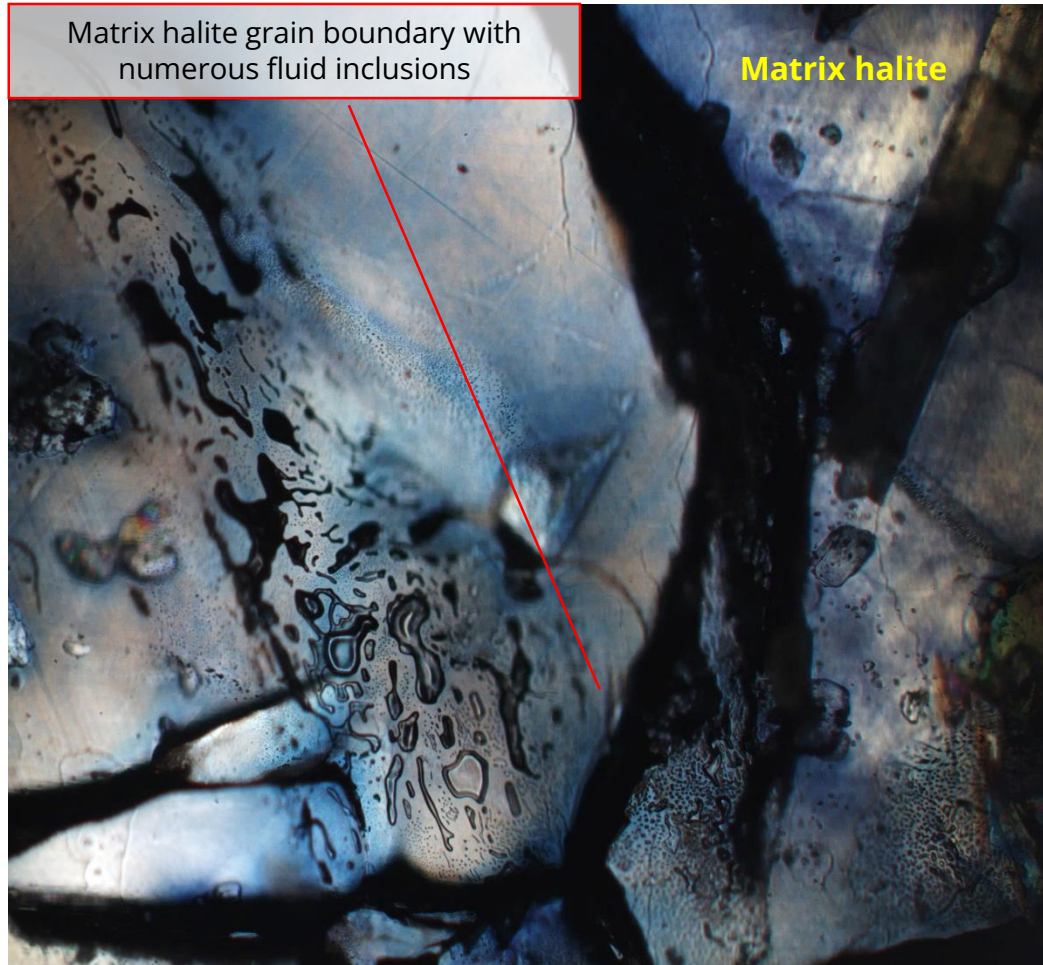
Tectonically-deformed twin: ZW-MaP_05g



500µm

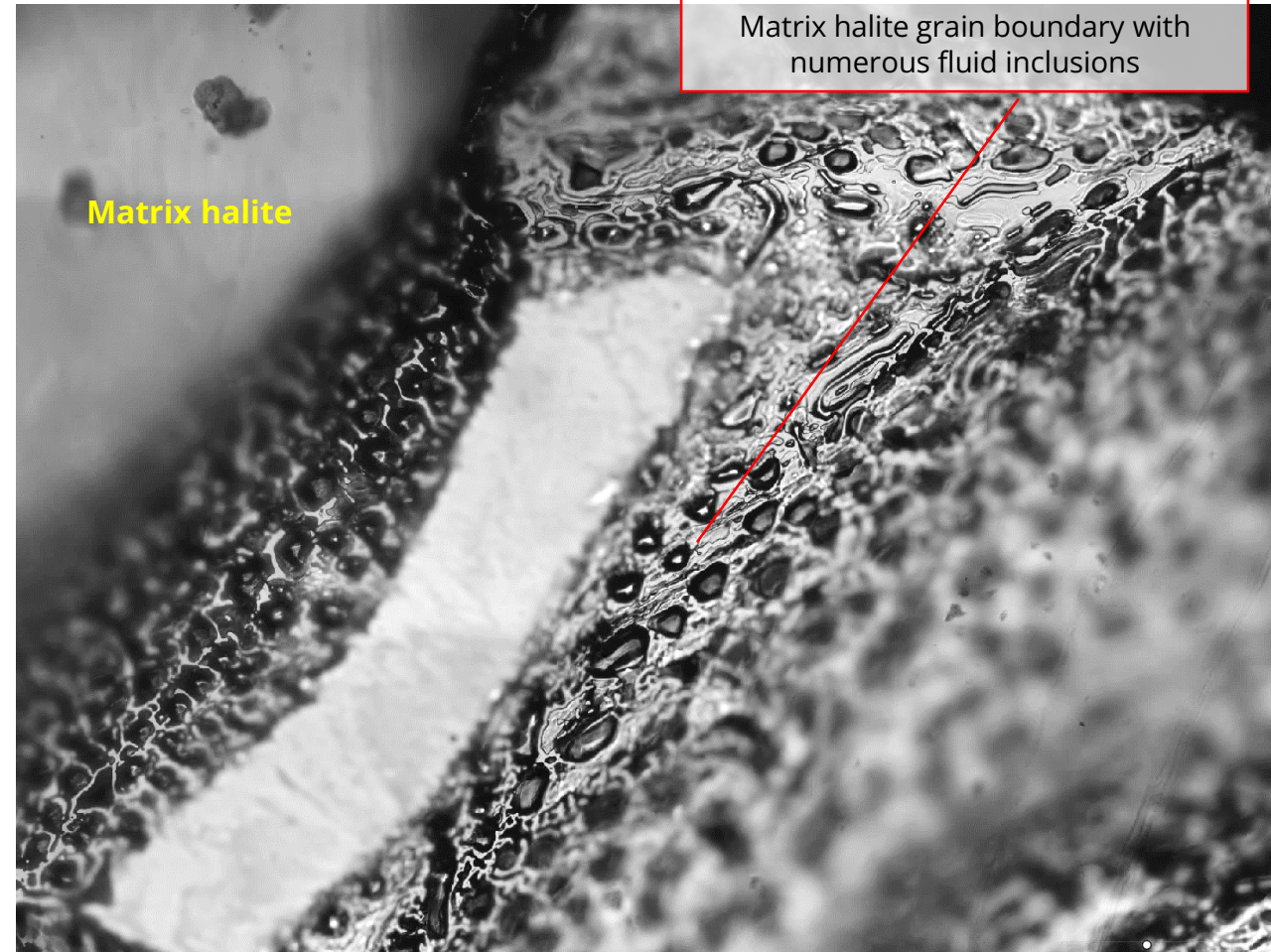
Lab-deformed twin: Zuidwending-3_TC4_MaP_42

Grain boundary structure: Zuidwending-3_TC4_MaP_42_3.0MPa



200µm

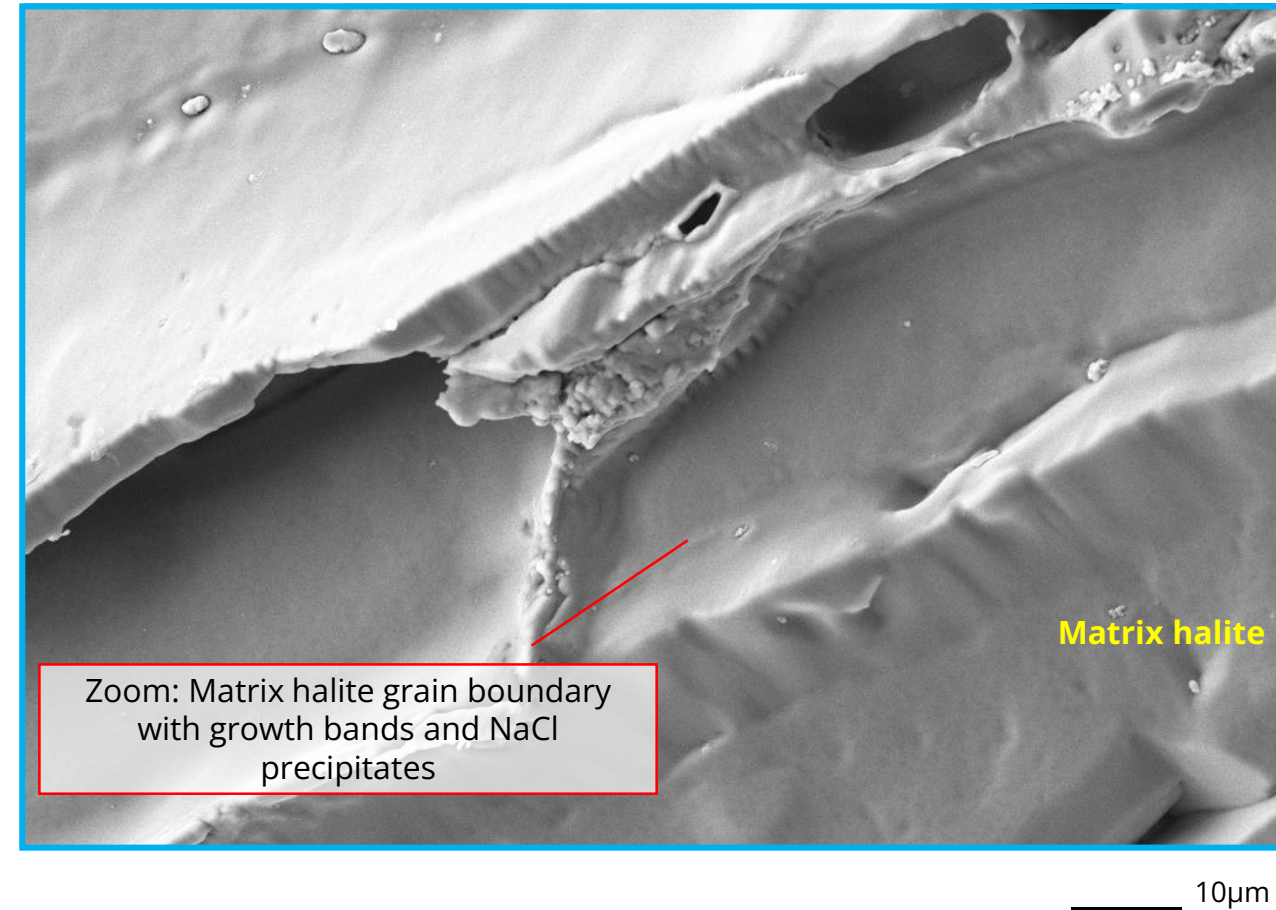
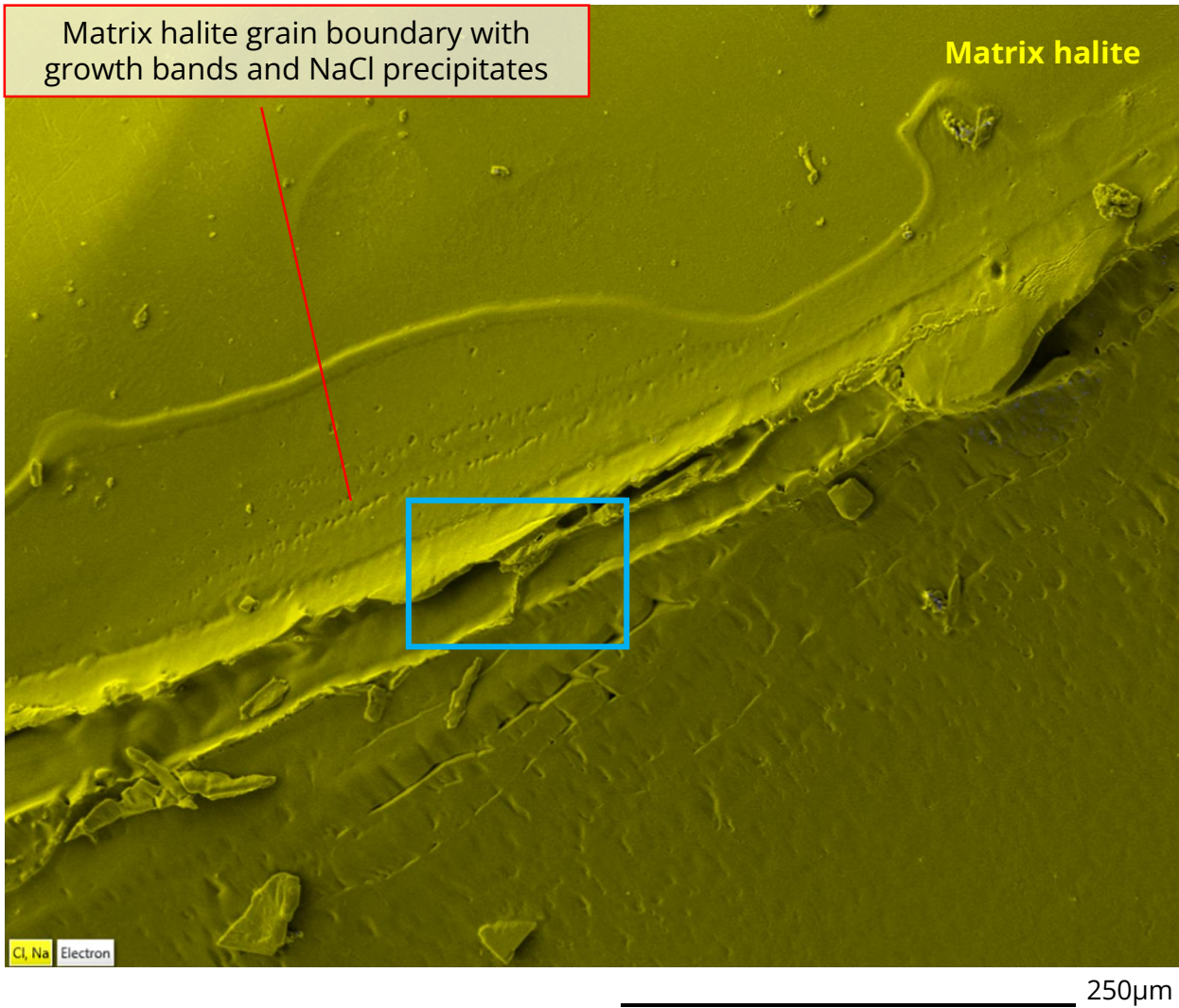
Tectonically-deformed twin: ZW-MaP_05g



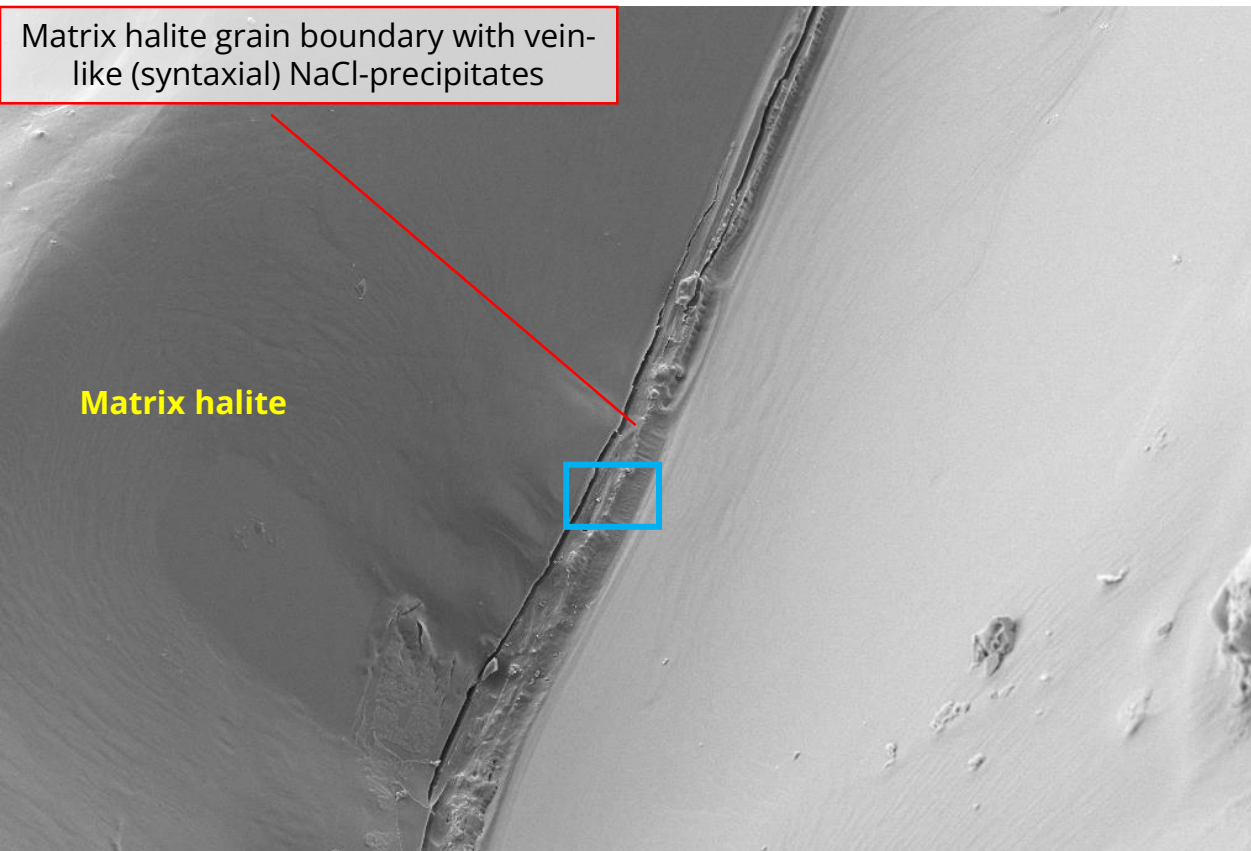
500µm

Lab-deformed twin: Zuidwending-3_TC4_MaP_42

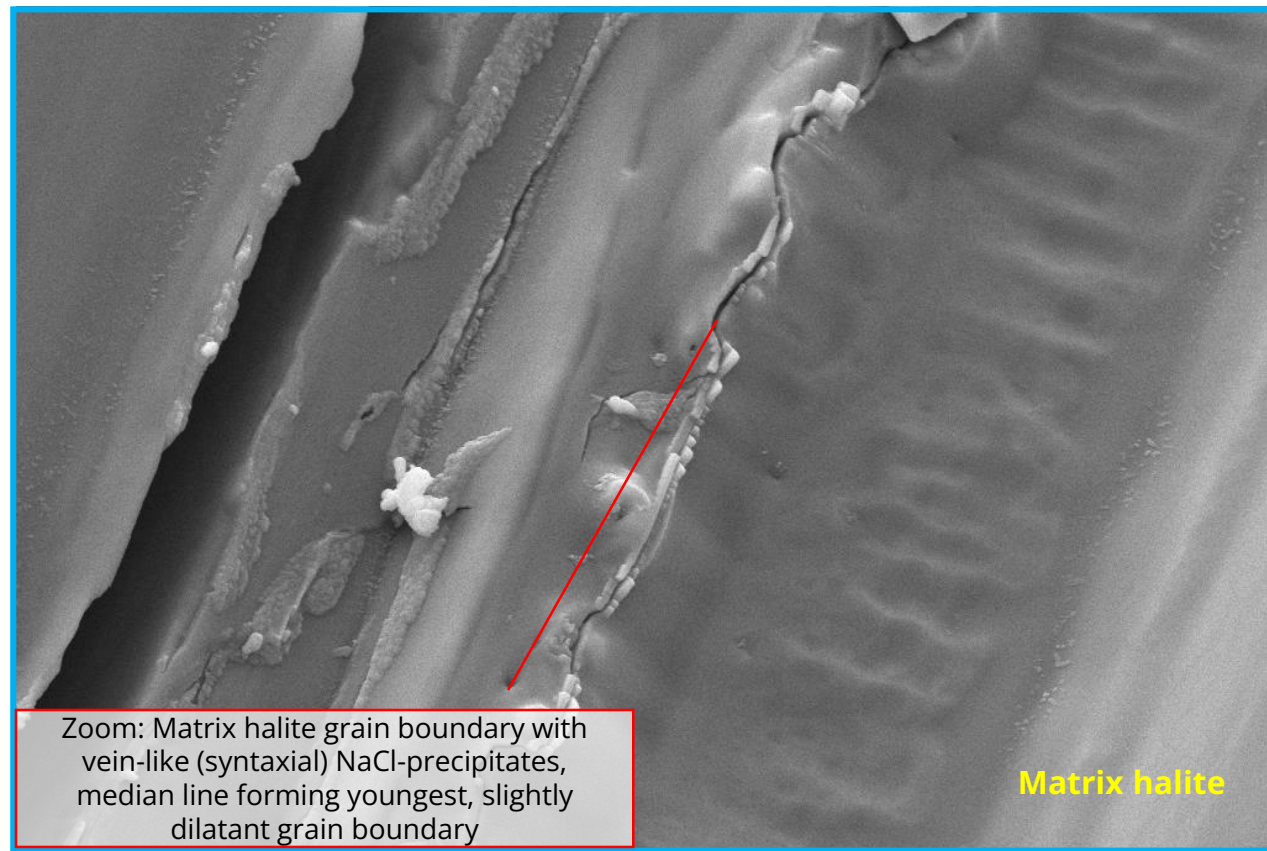
Grain boundary structure: **Zuidwending-3_TC4_MaP_42_3.0MPa**



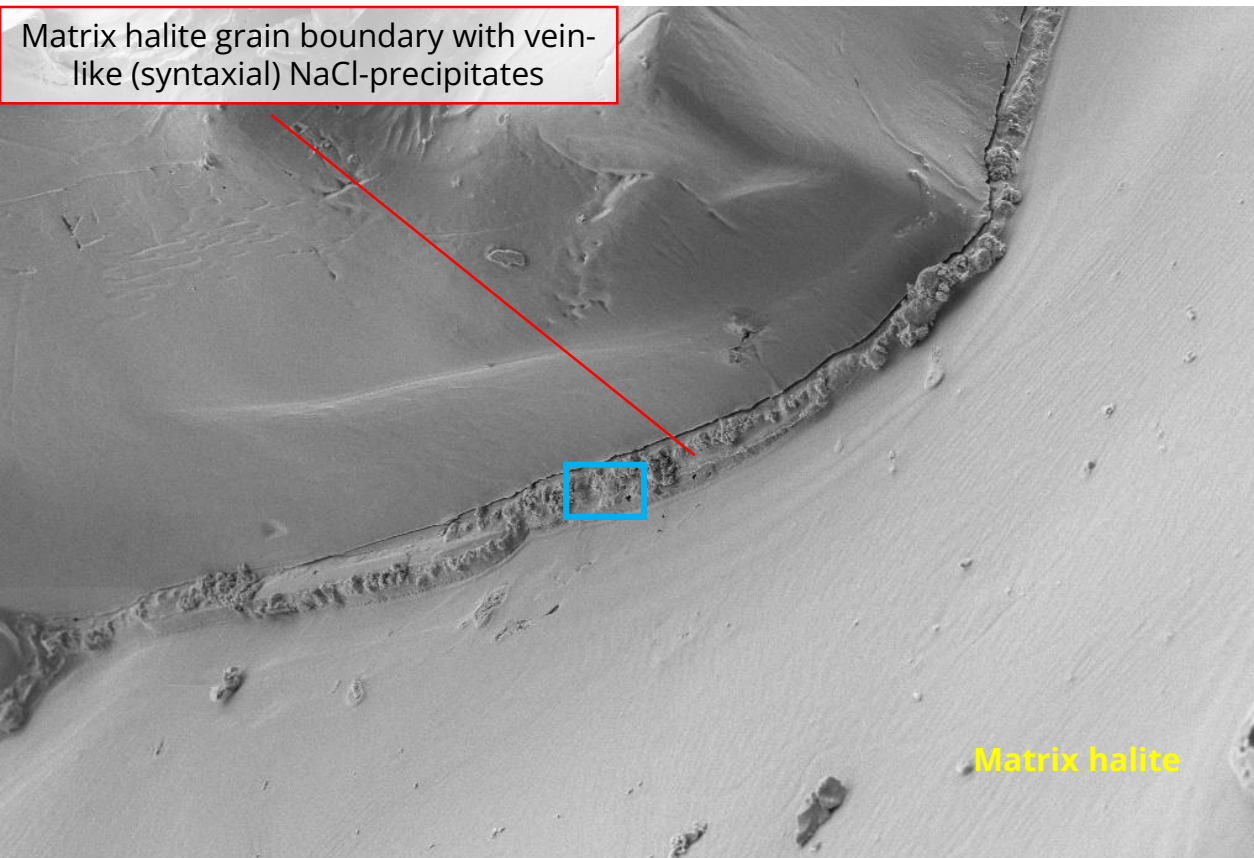
Grain boundary structure: Zuidwending-3_TC4_MaP_42_3.0MPa



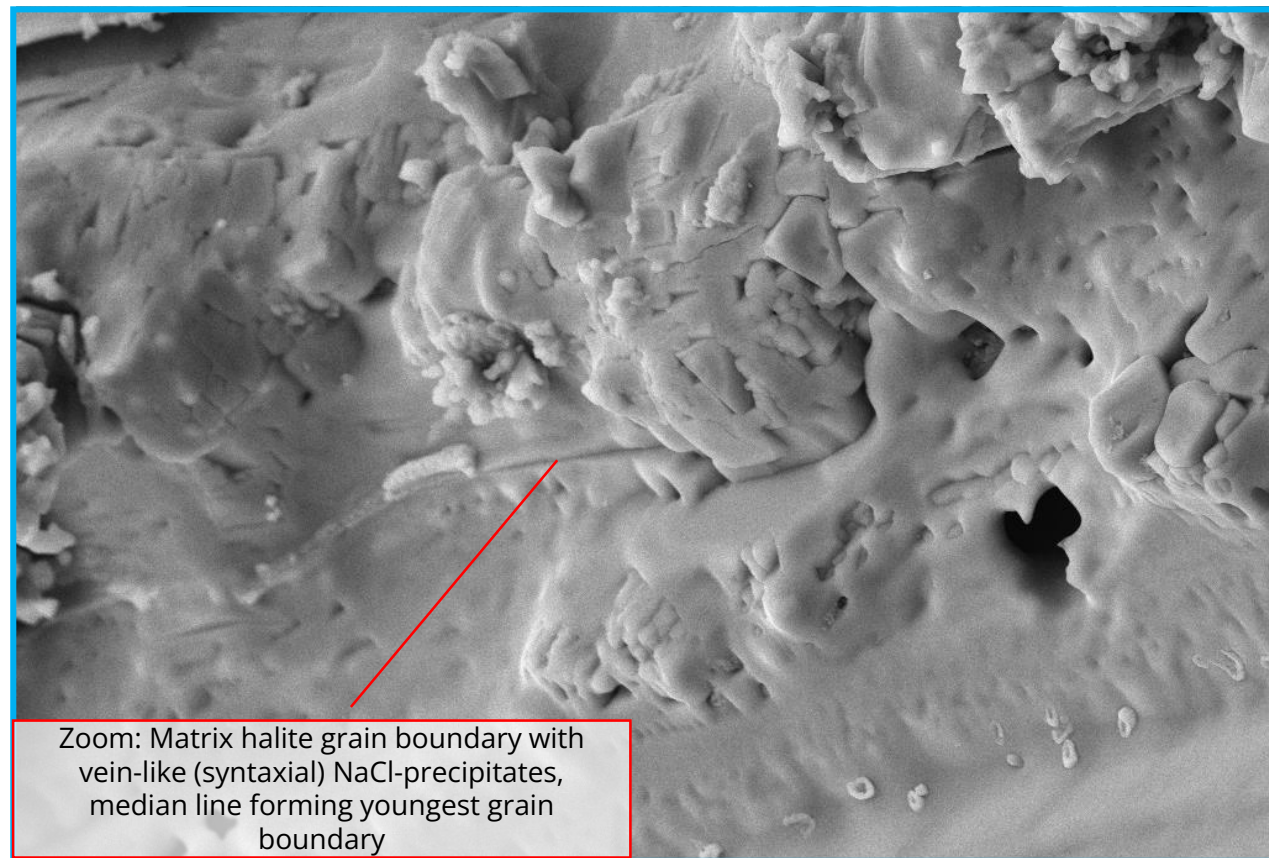
— 20μm



— 2μm



100µm



2µm

Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.

Core piece photography: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**

Reflected light



Transmitted light



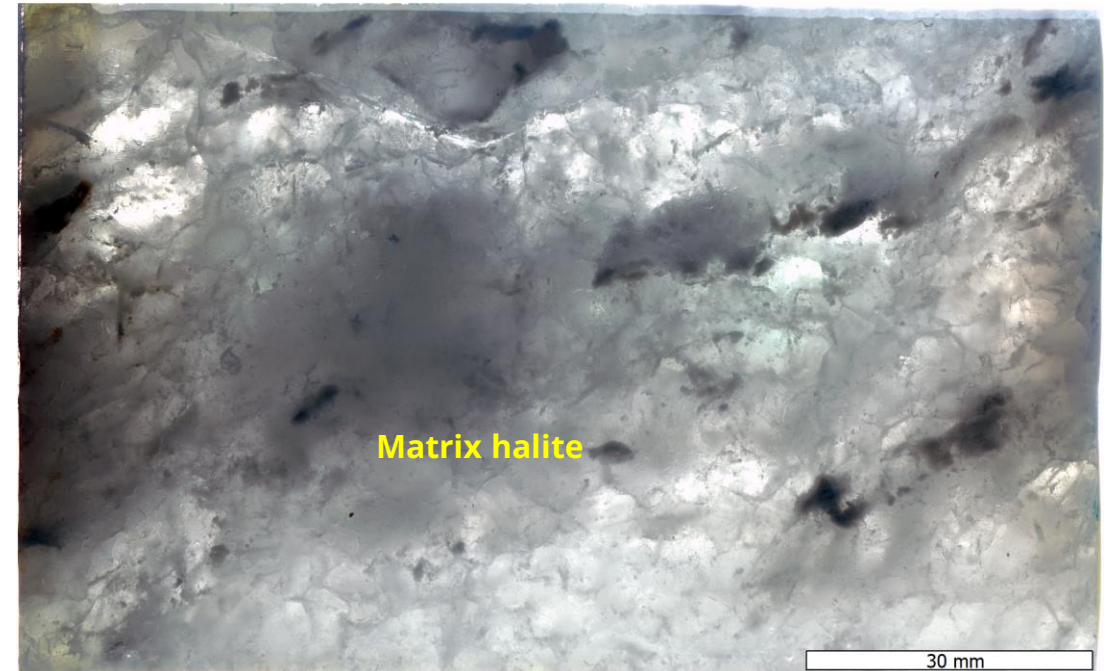
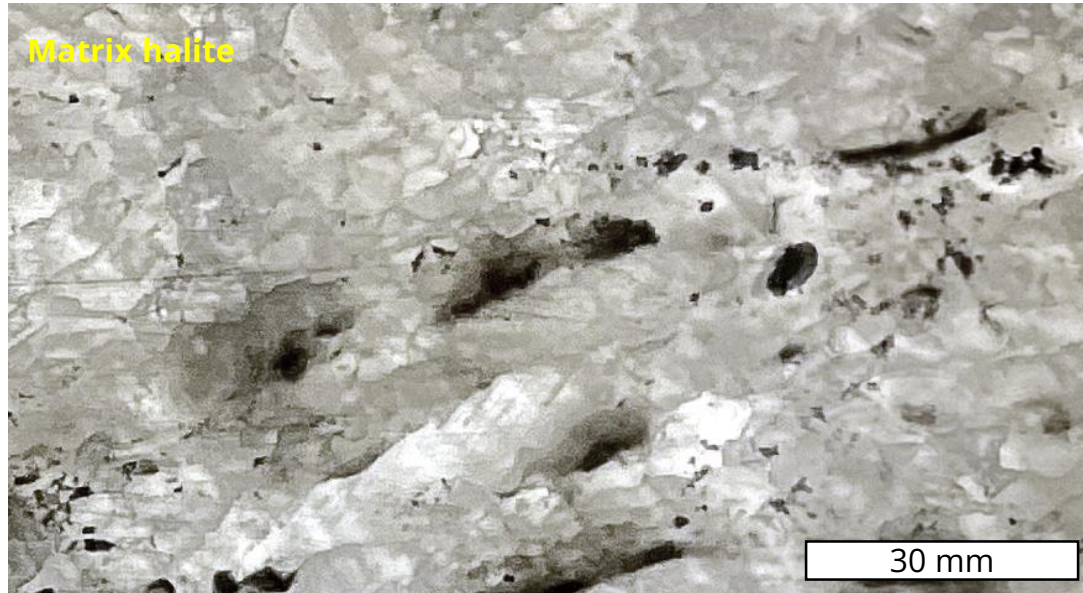
Transmitted light
(cut-side)



Reflected light
(cut-side)



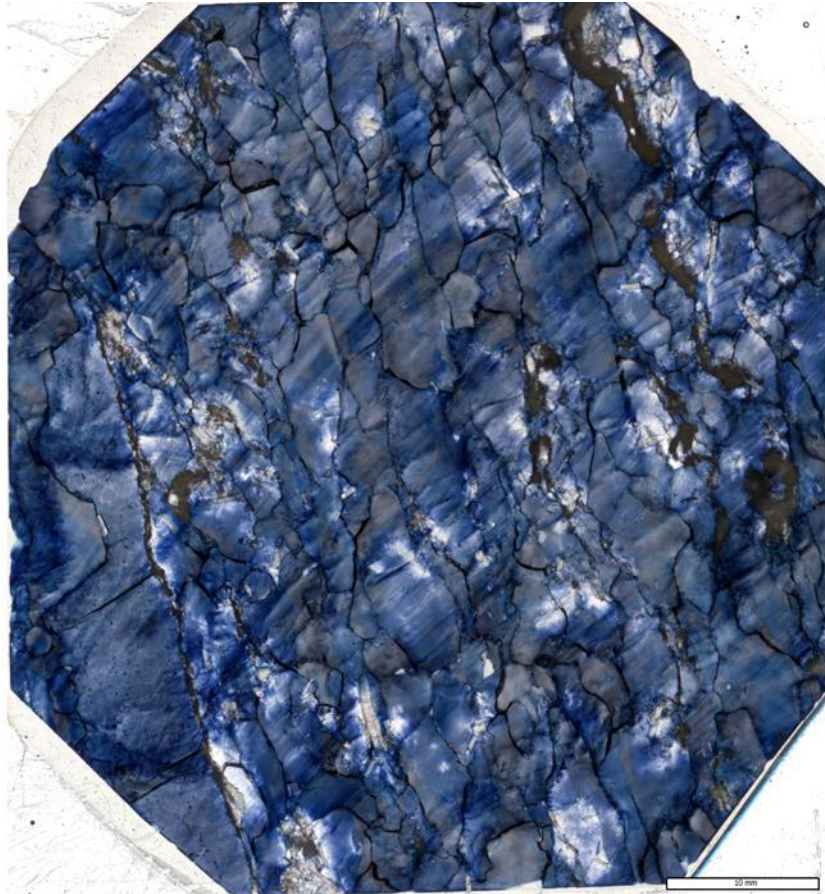
Core piece slab photo: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**



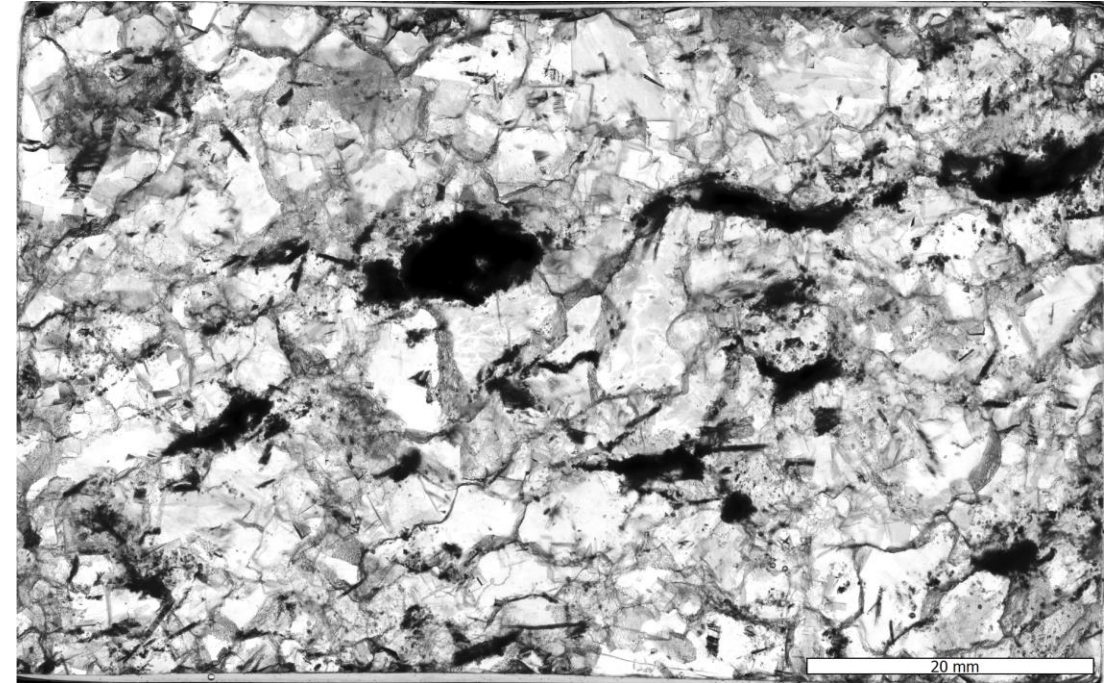
Tectonically-deformed twin: ZW-MaP_05g

Lab-deformed twin: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def

Thin section in transmitted light: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**

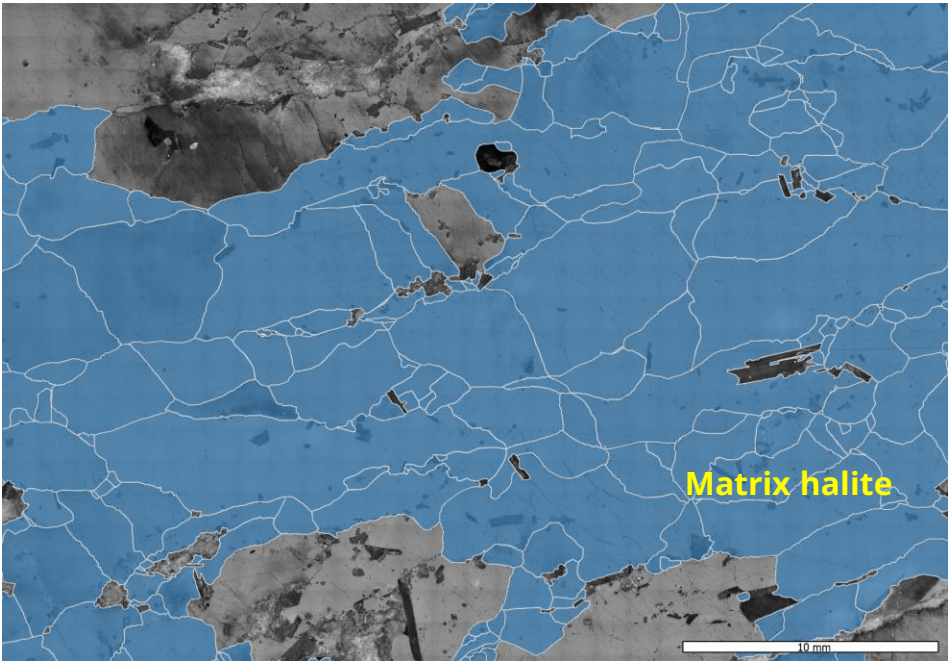


Tectonically-deformed twin: ZW-MaP_05g

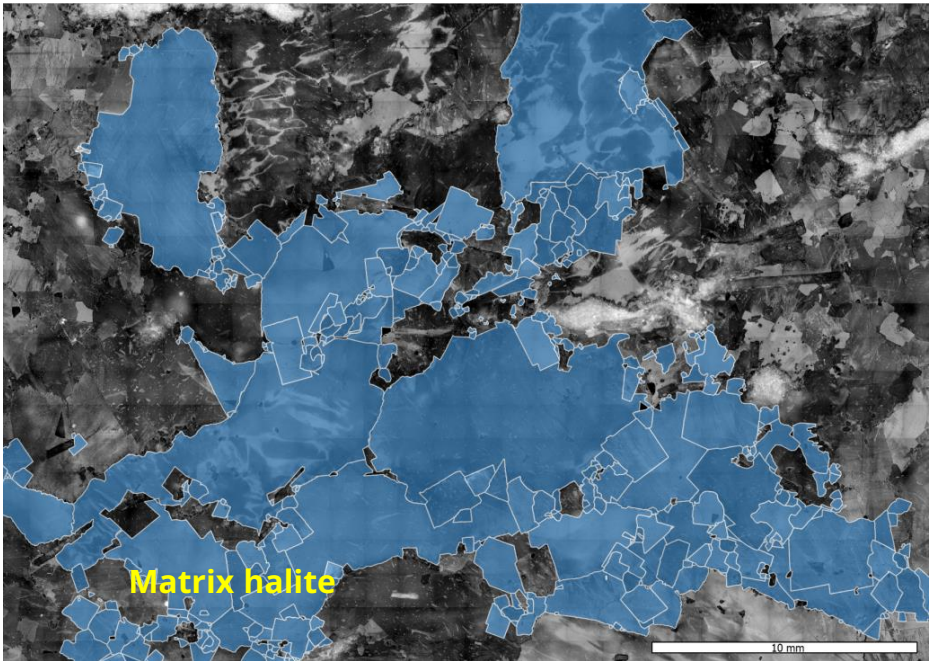


Lab-deformed twin: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def

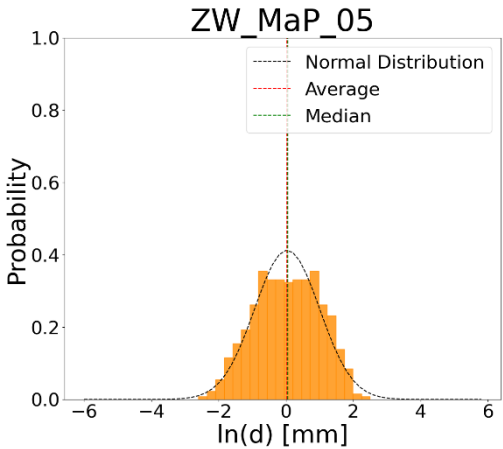
Grain size (d) distribution: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**



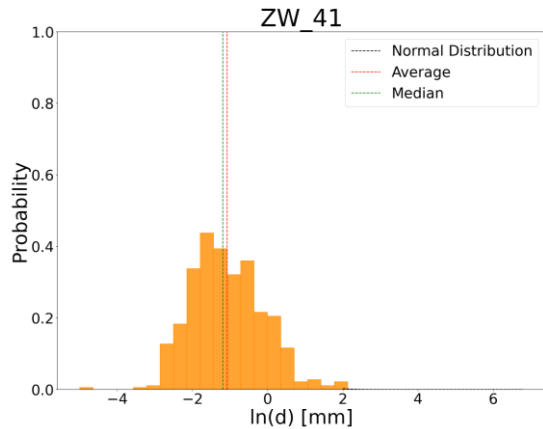
Tectonically-deformed twin: ZW-MaP_05g



Lab-deformed twin: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def

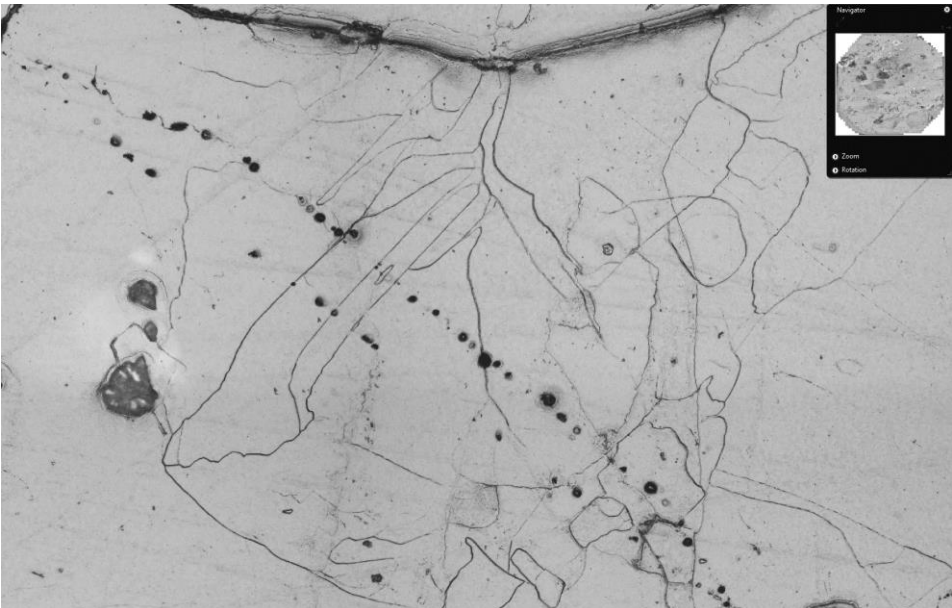


	d [mm]	ln(d)[mm]
Number of grains	507	507
Min [mm]	0.07	-2.61
Max [mm]	12.18	2.50
Median [mm]	1.05	0.05
Average [mm]	1.60	0.03
Std_deviation [mm]	1.54	0.97
Mode [mm]	1.51	0.41



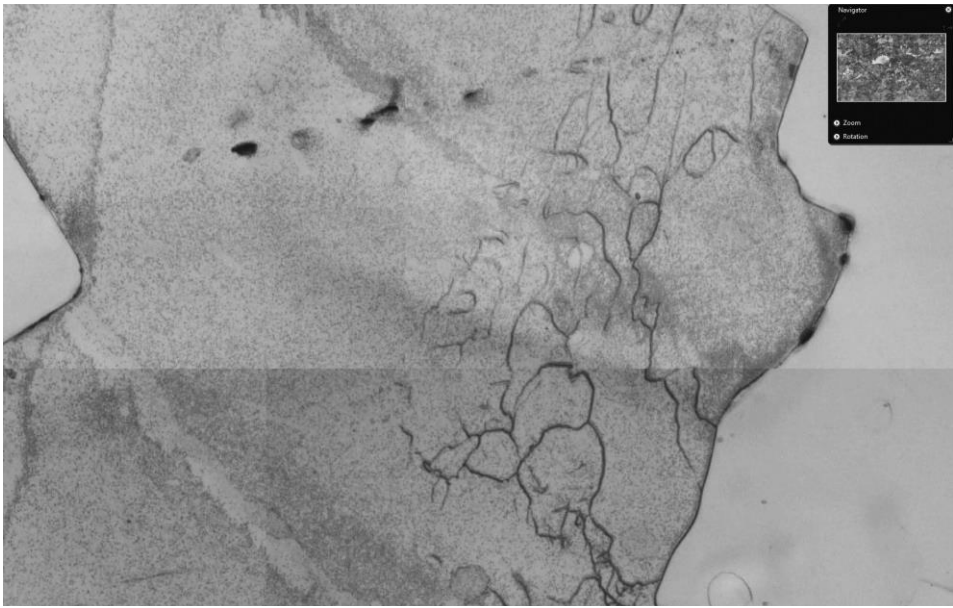
	d [mm]	ln(d)[mm]
Number of grains	506	506
Min [mm]	0.01	-5.14
Max [mm]	8.44	2.13
Median [mm]	0.30	-1.20
Average [mm]	0.57	-1.08
Std_deviation [mm]	0.84	0.97
Mode [mm]	1.80	0.59

Subgrain size (D) distribution: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**



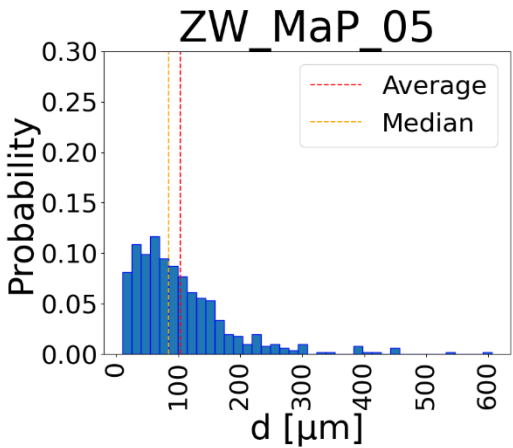
100µm

Tectonically-deformed twin: ZW-MaP_05g

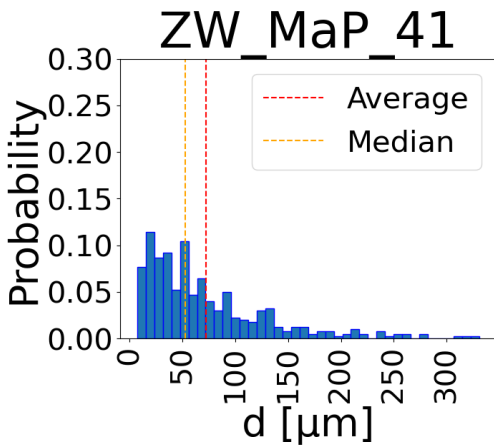


50µm

Lab-deformed twin: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def

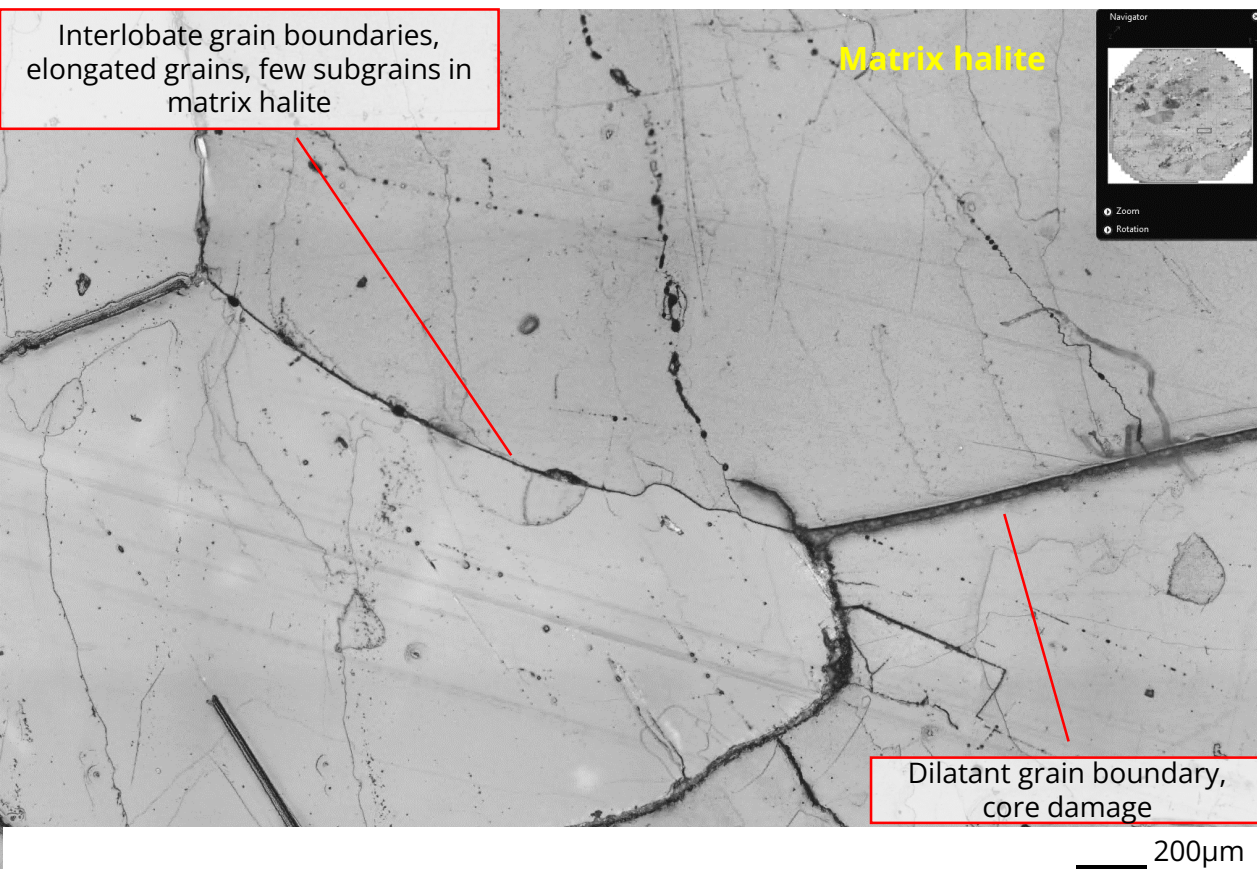


	d [µm]	ln(d)[µm]
Number of grains	505	505
Min [µm]	9.93	2.30
Max [µm]	605.78	6.41
Median [µm]	84.39	4.44
Average [µm]	103.75	4.37
Std_deviation [µm]	81.55	0.77
Mode [µm]	64.63	4.17

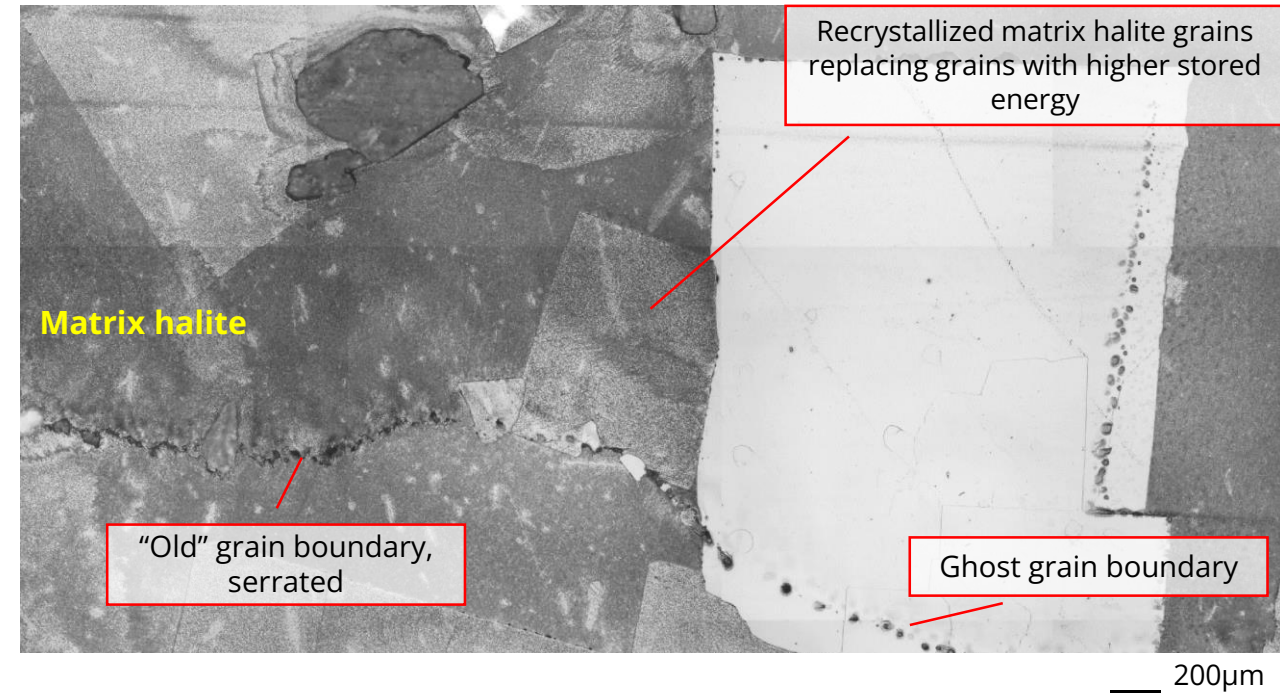


	d [µm]	ln(d)[µm]
Number of grains	402	402
Min [µm]	7.10	1.96
Max [µm]	330.86	5.80
Median [µm]	52.65	3.96
Average [µm]	71.95	3.95
Std_deviation [µm]	59.86	0.83
Mode [µm]	117.40	4.77

Texture: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.

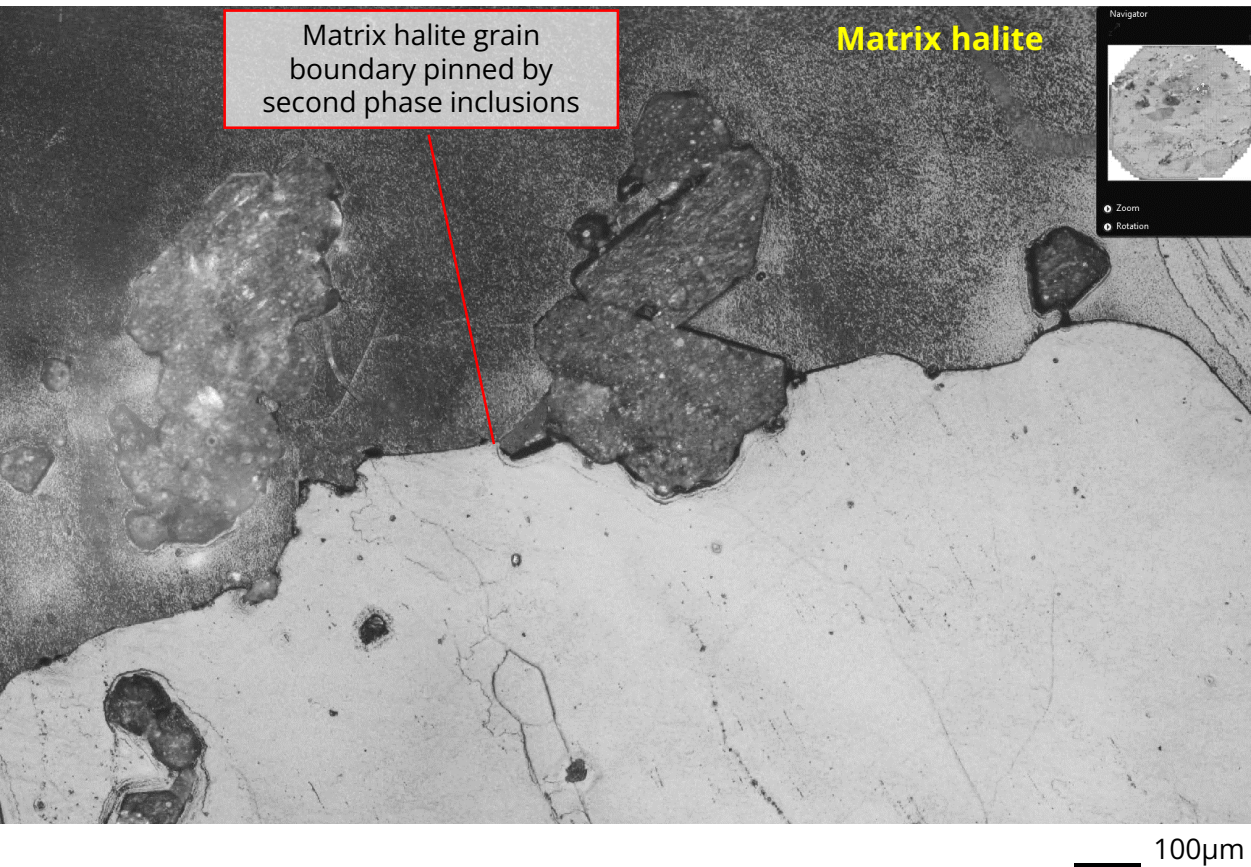


Tectonically-deformed twin: ZW-MaP_05g

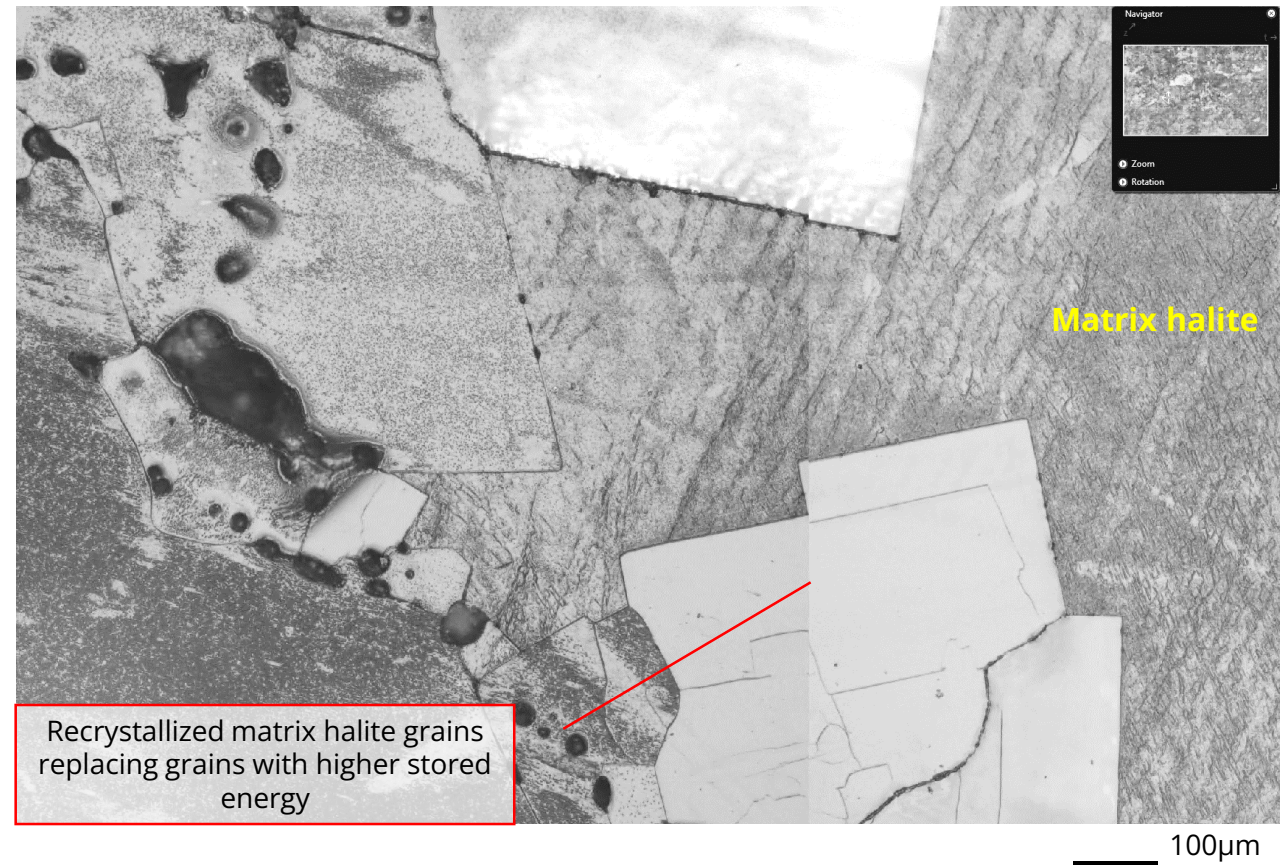


Lab-deformed twin: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def

Texture: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.

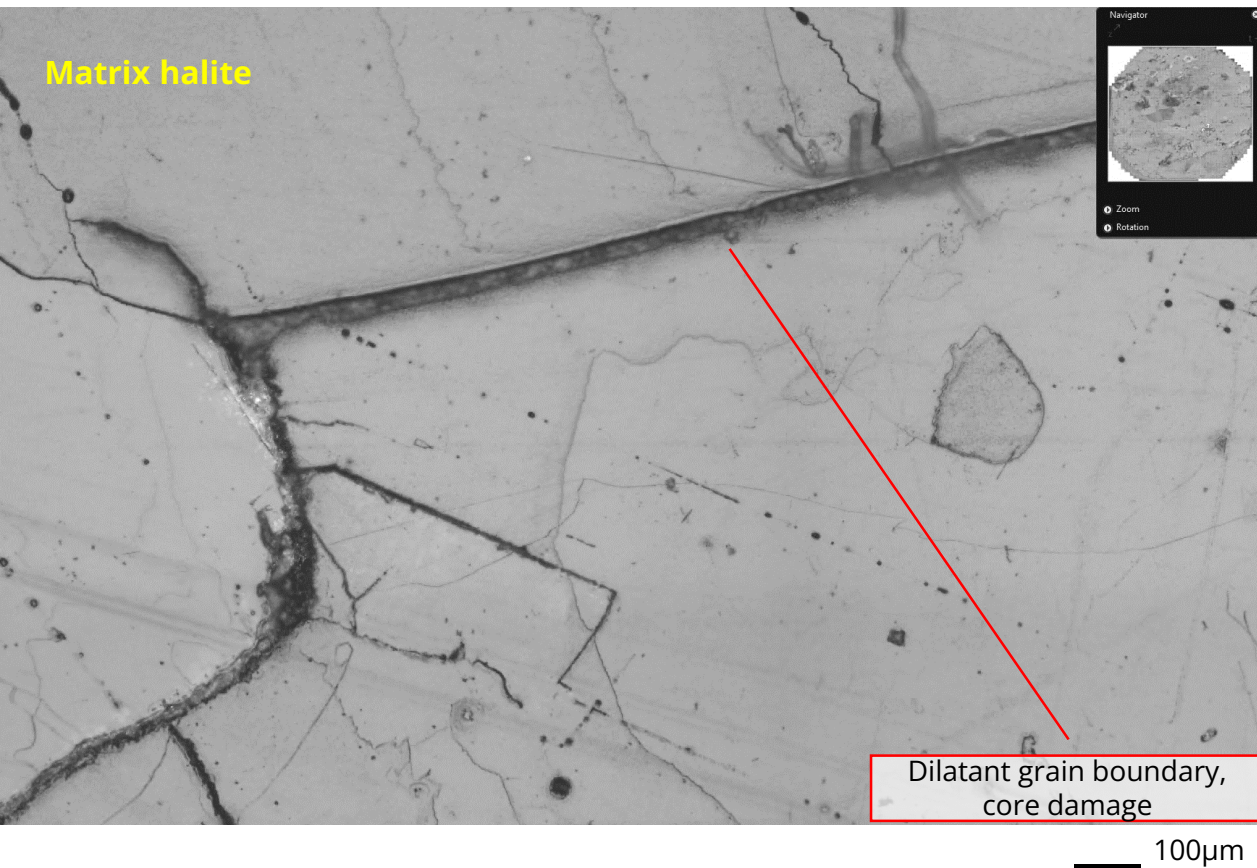


Tectonically-deformed twin: ZW-MaP_05g

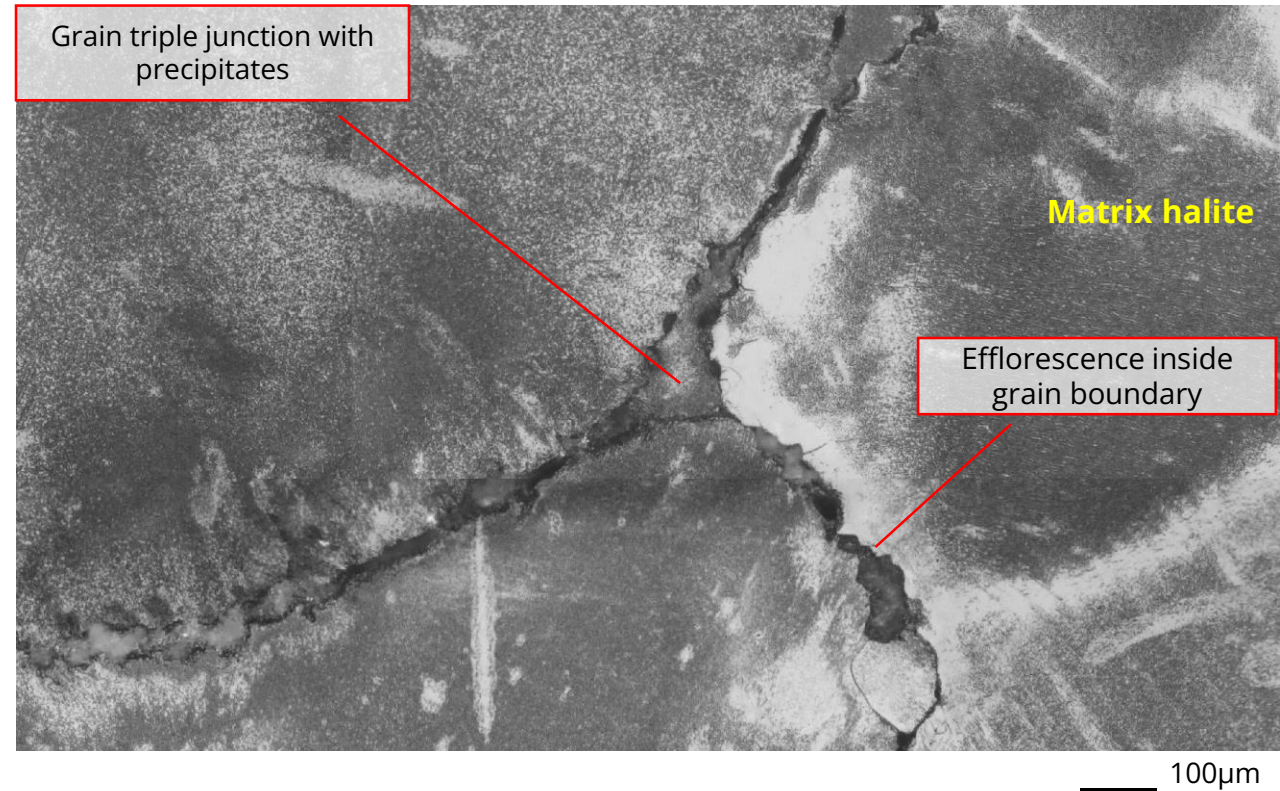


Lab-deformed twin: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def

Texture: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.

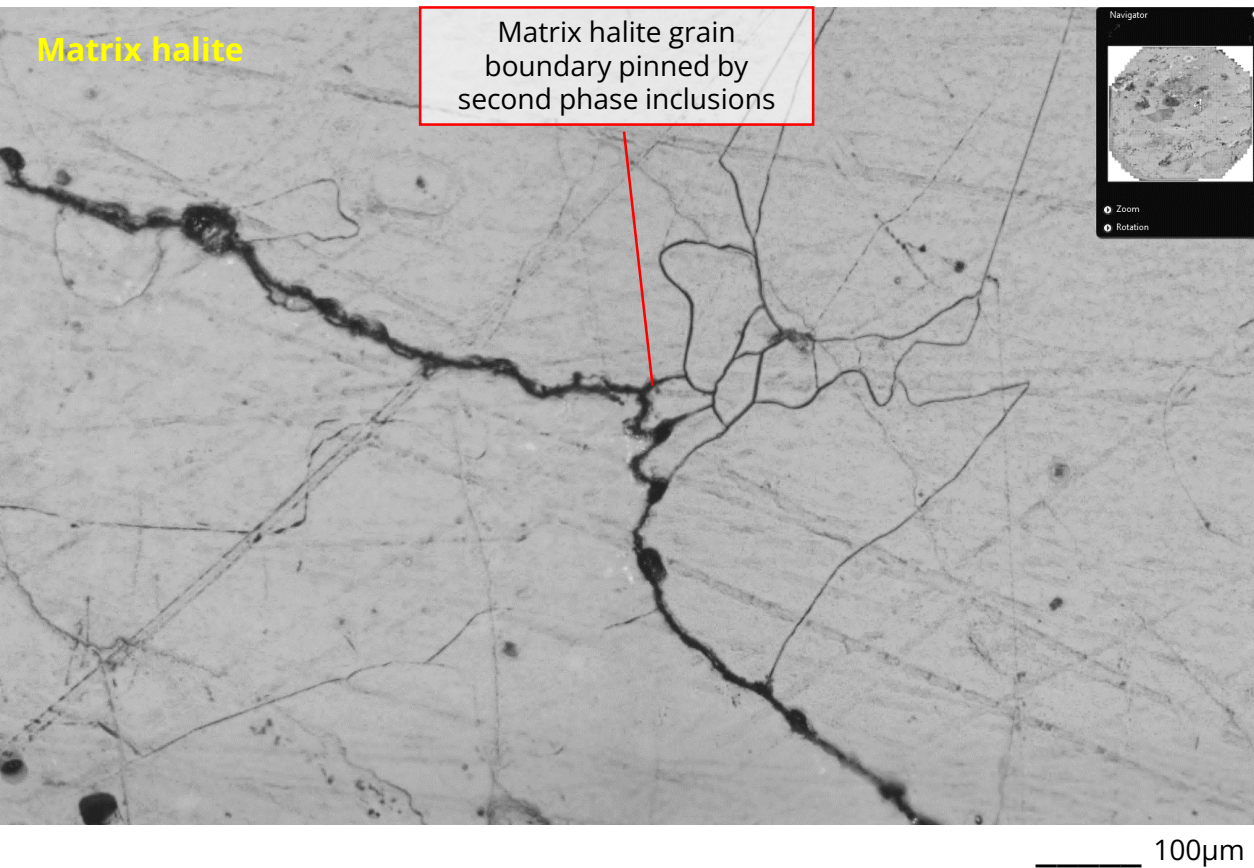


Tectonically-deformed twin: ZW-MaP_05g

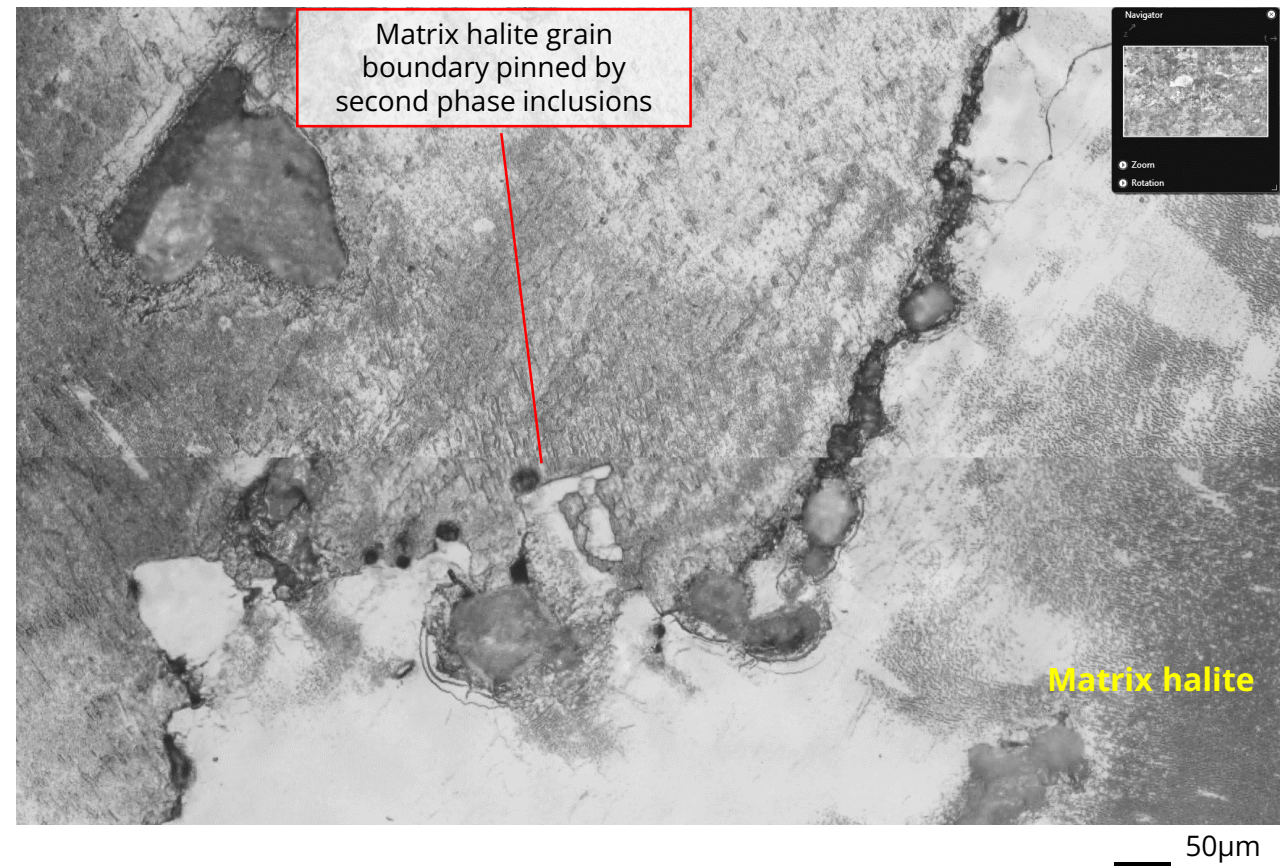


Lab-deformed twin: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def

Grain boundary structure: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.

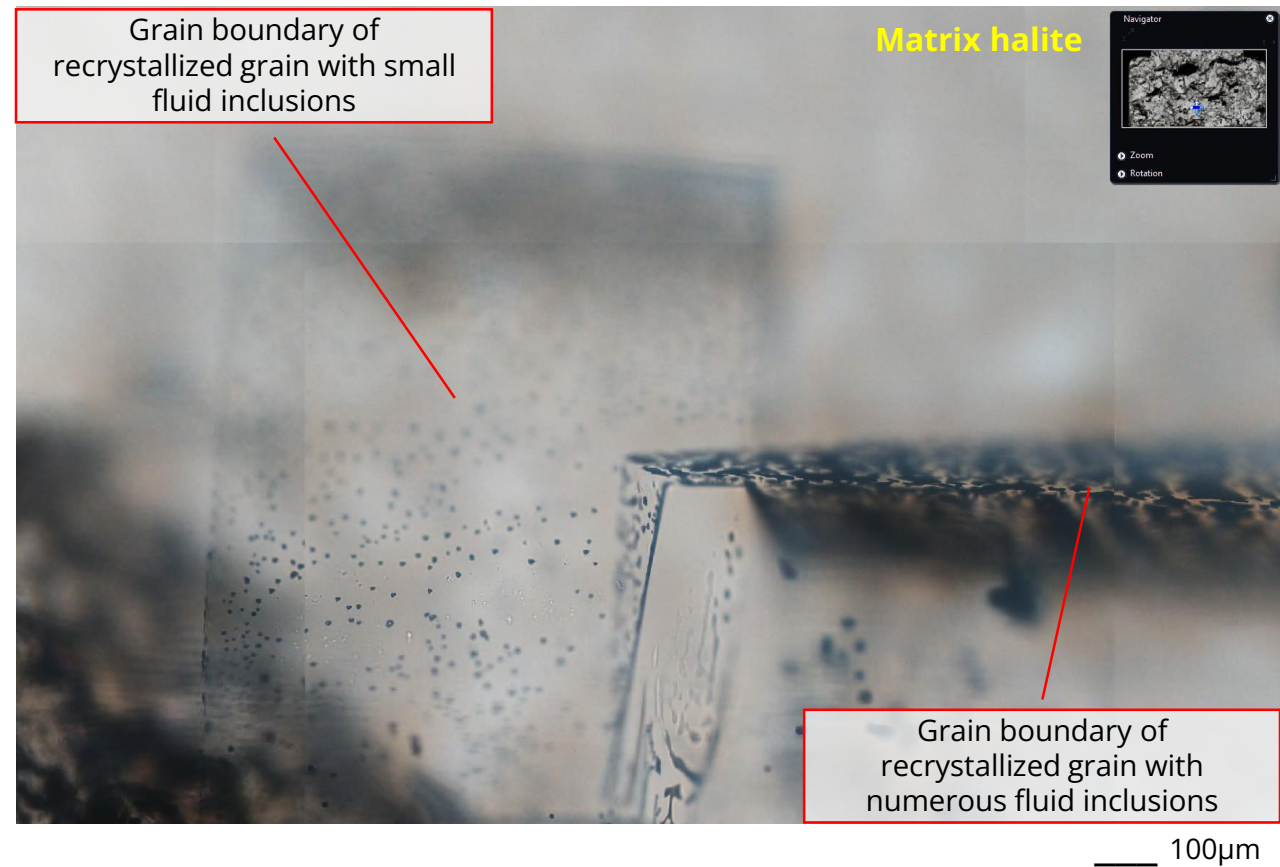
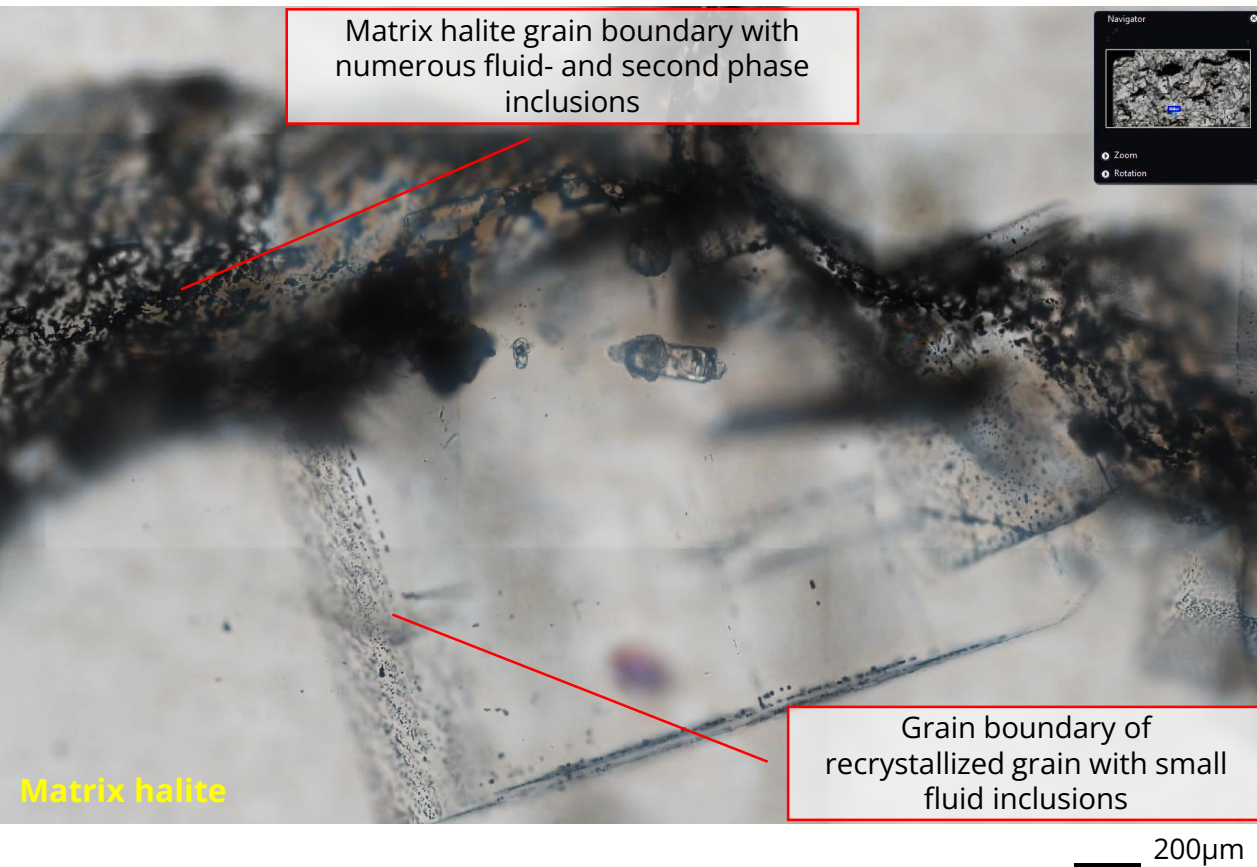


Tectonically-deformed twin: ZW-MaP_05g

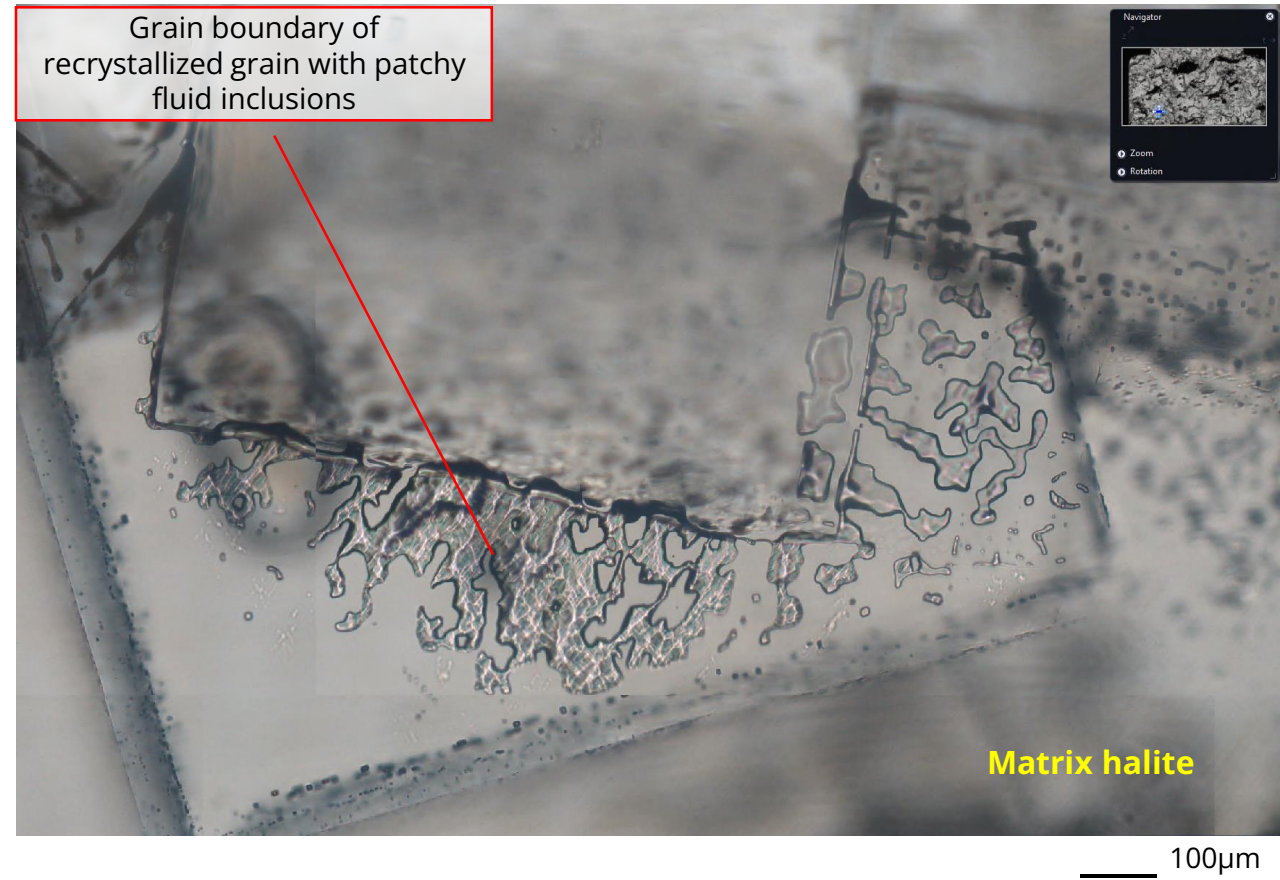
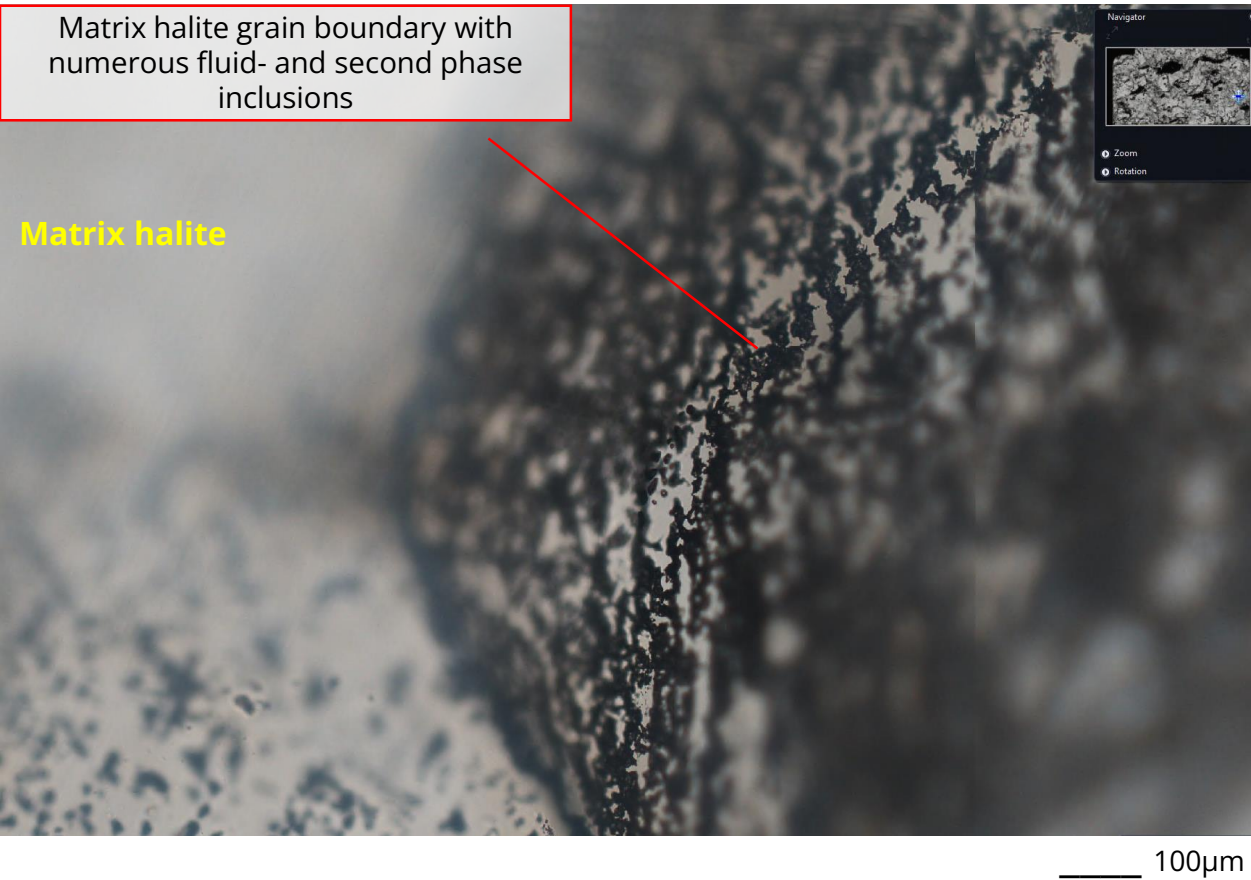


Lab-deformed twin: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.

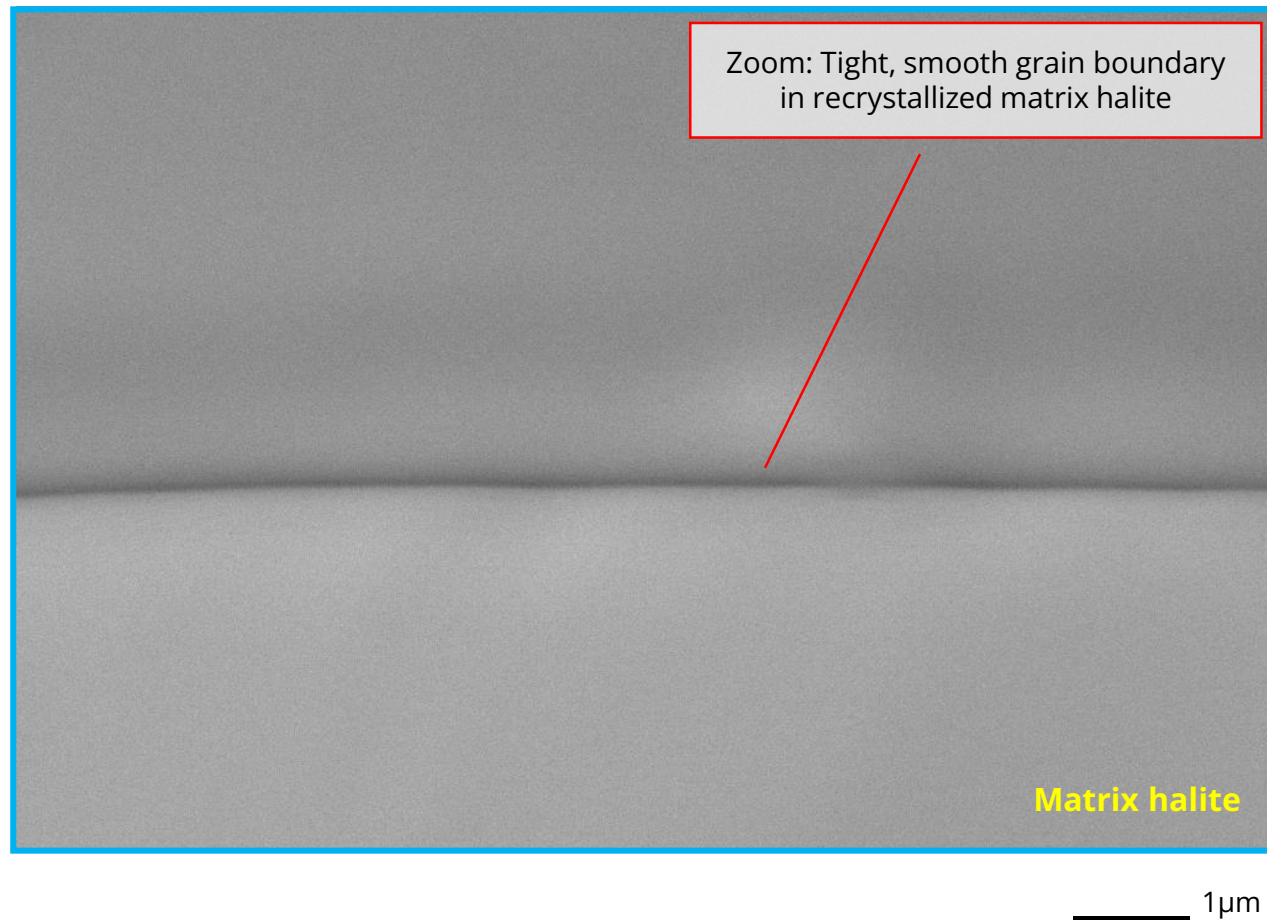
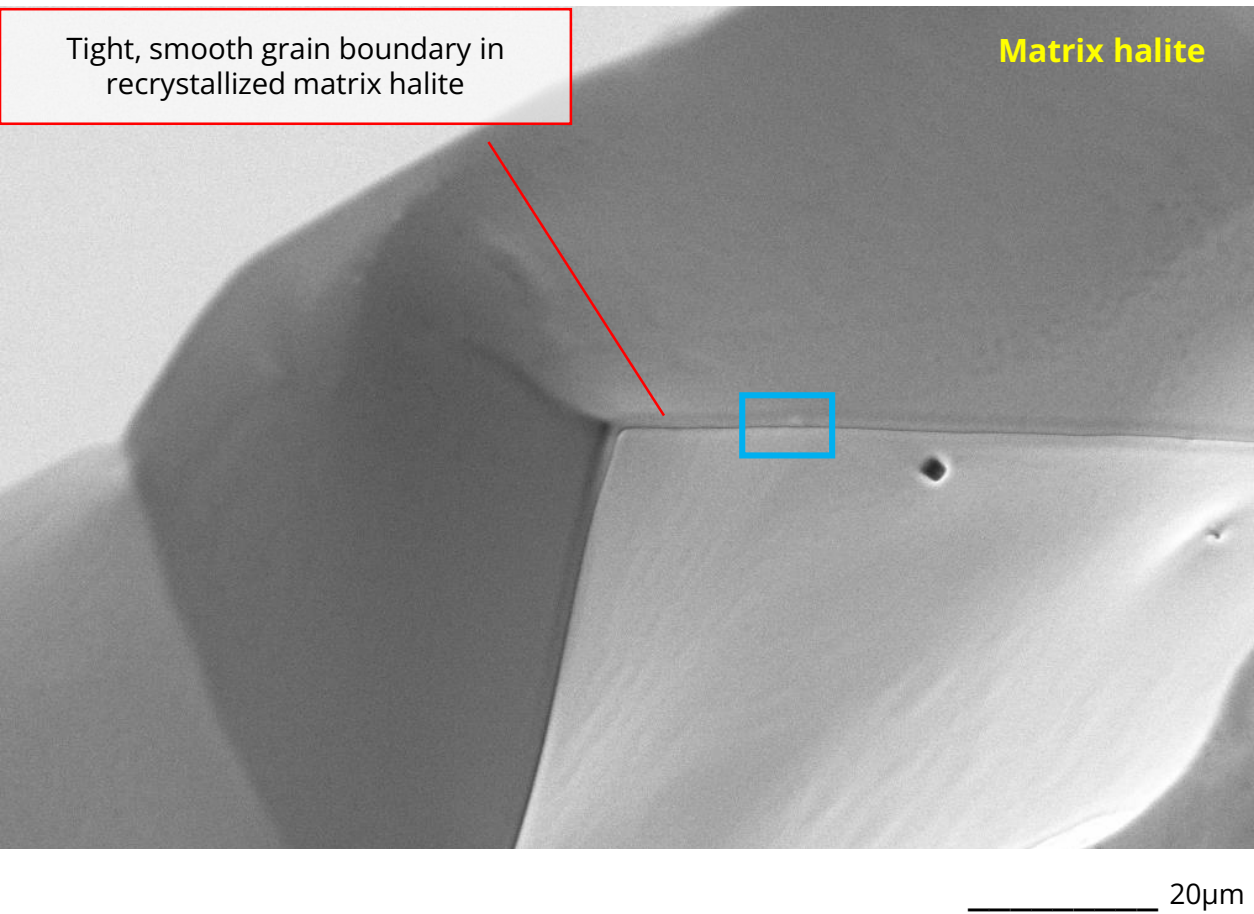
Grain boundary structure: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**



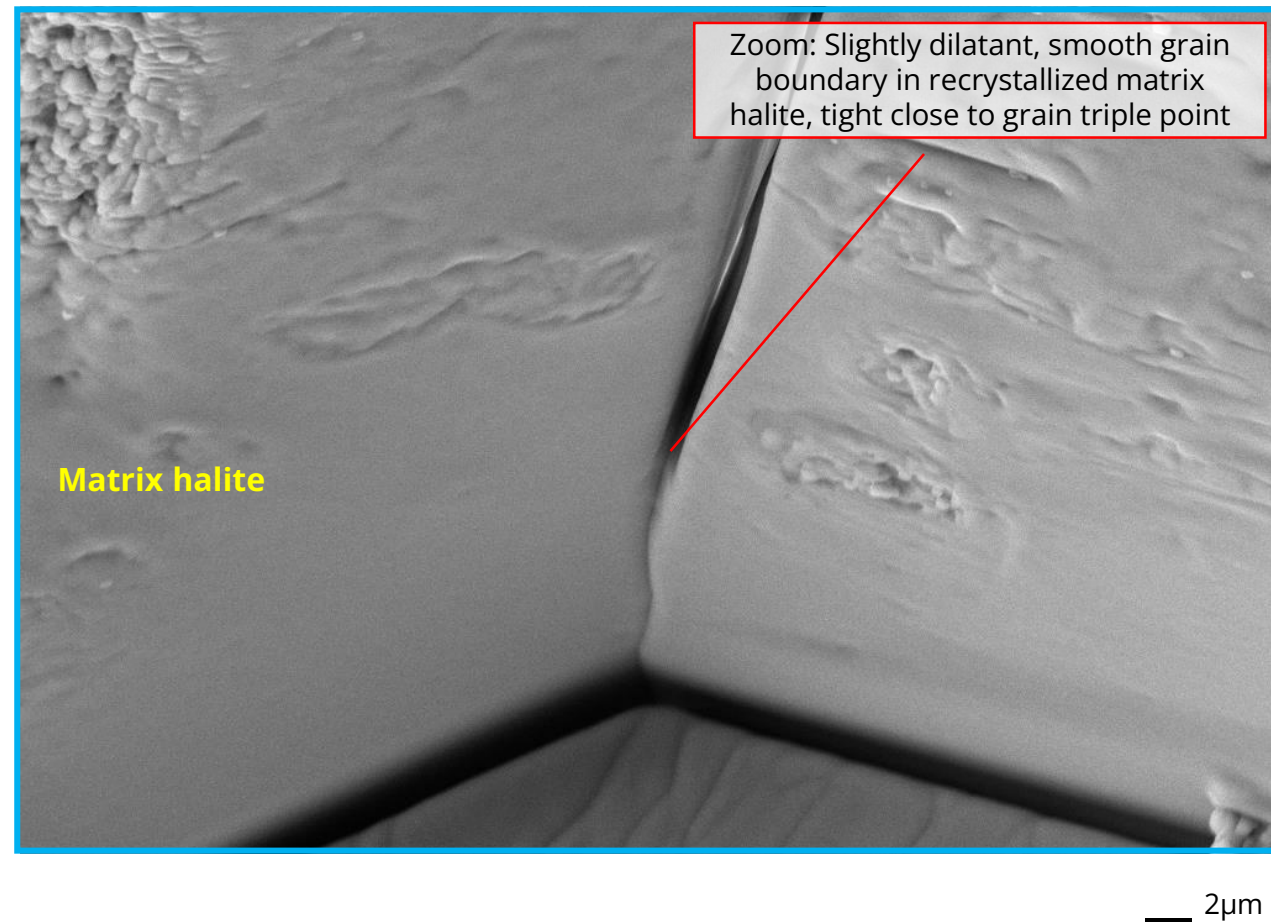
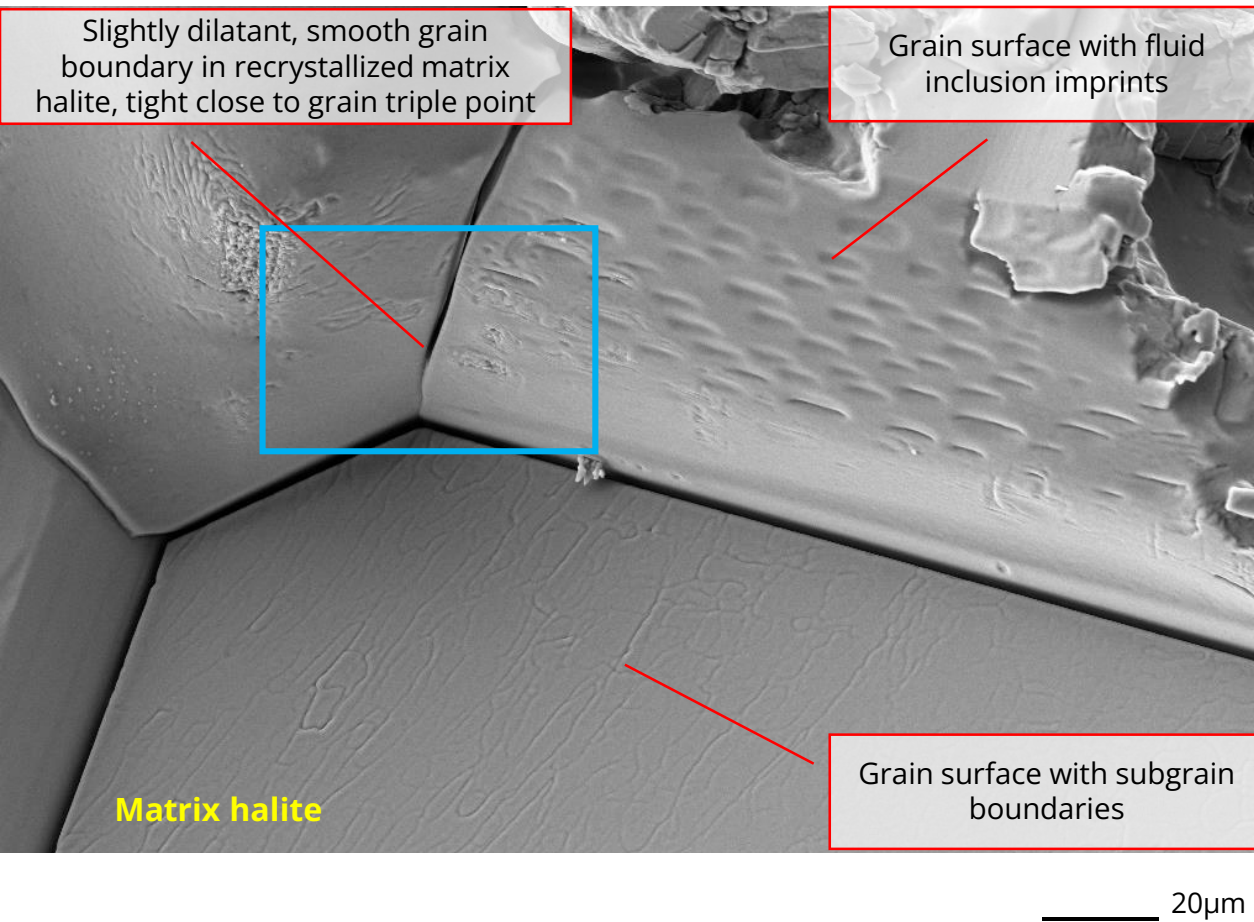
Grain boundary structure: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**



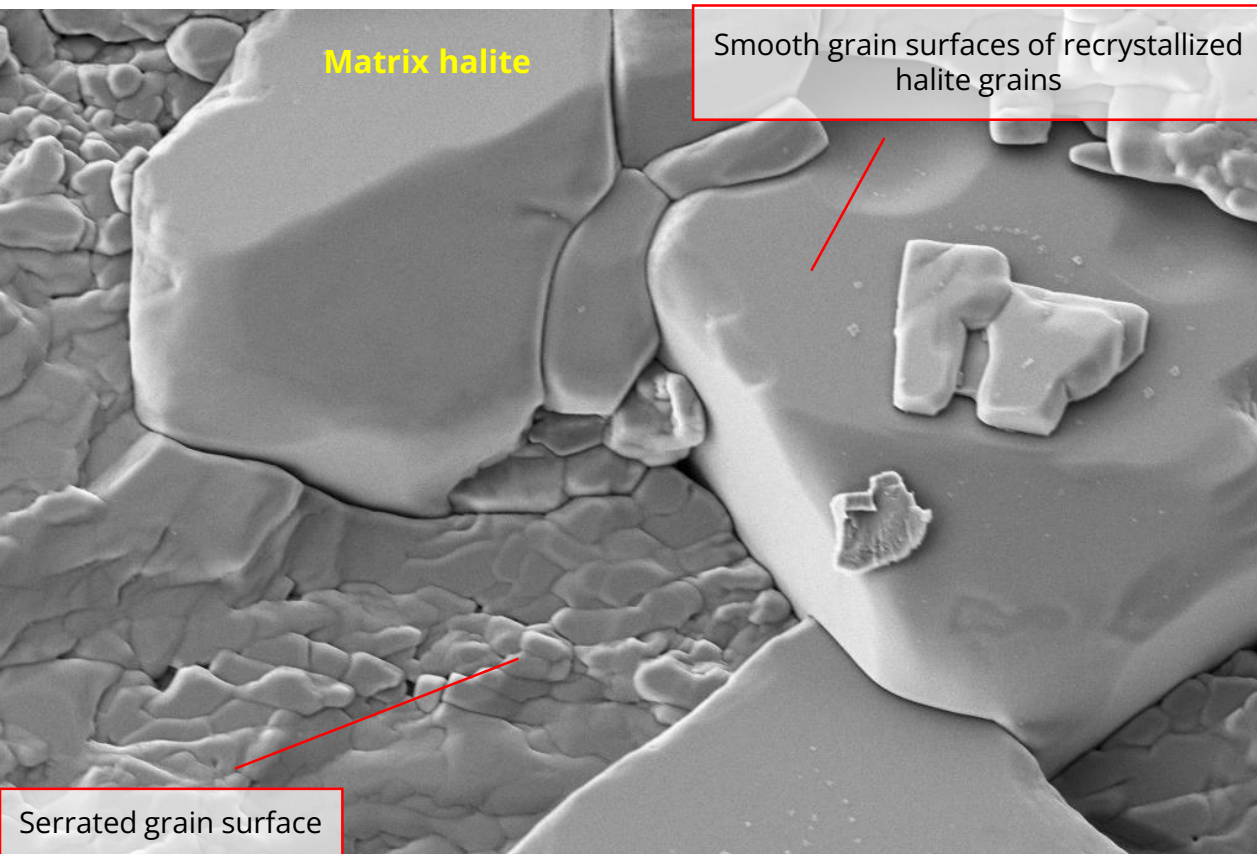
Grain boundary structure: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**



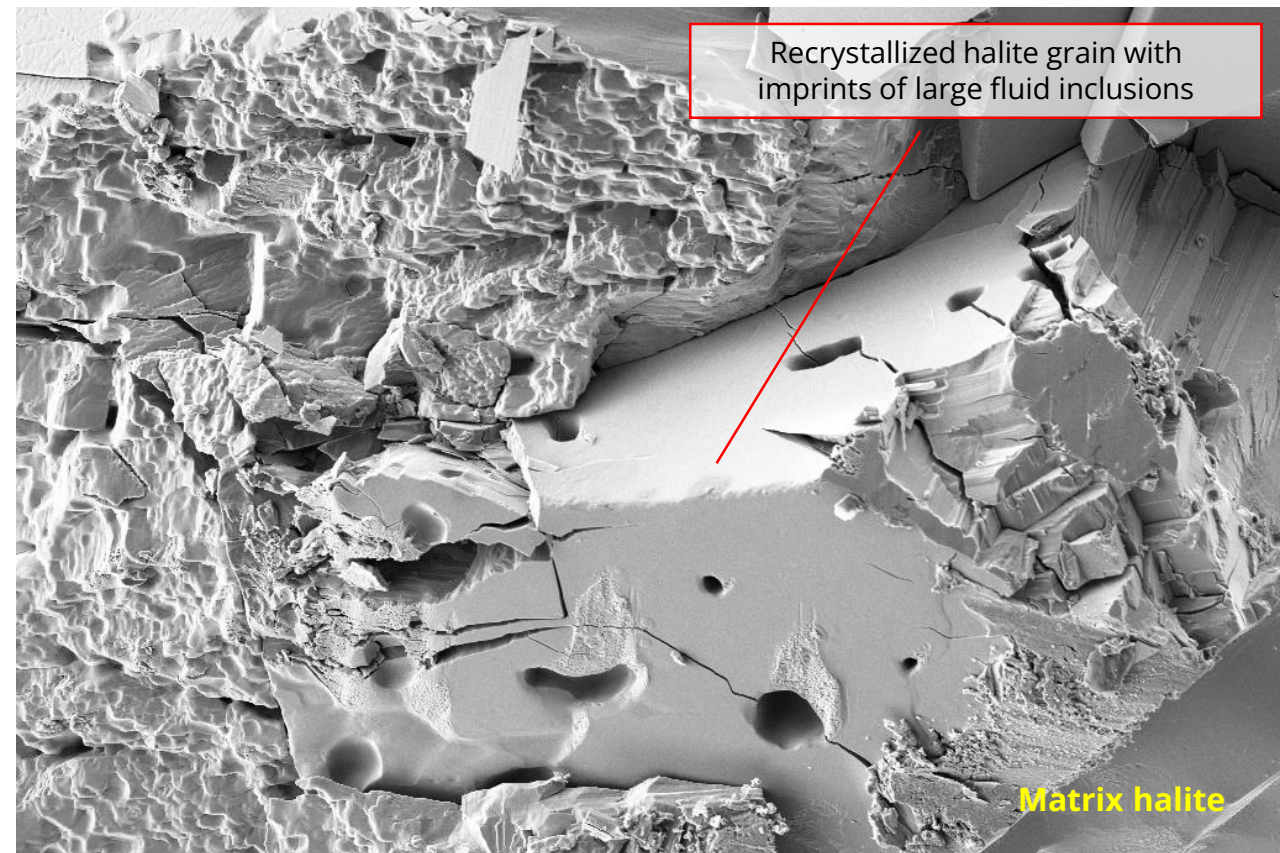
Grain boundary structure: **Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.**



Grain boundary structure: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.



20µm



100µm

Grain boundary structure: Zuidwending-4_TC3_MaP_41_0.6MPa_pre-def.

