Creep behaviour of bischofite, carnallite and mixed bischofite-carnallite-halite salt rock at in situ conditions

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1. Background, problem and aim:

- Salt deposits at Veendam (NL) contain bischofite ($MgCl_2 6H_2O$), carnallite ($KMgCl_3 6H_2O$) and halite (NaCl) that exist in layers and are extracted from the subsurface by solution mining in underground caverns;
- Abandonment of the caverns causes the wall rock to flow inward due to overburden stress, which in turn results in subsidence at the surface;
- The physics of flow of these salts is of utmost importance to understand for a reliable prediction of surface subsidence, but no yet fully known;
- Main aim: constitutive flow laws than can be applied at real *in situ* conditions.

2. Material and experiments:

- Polycrystalline cylindrical samples from as received cores, bischofite, carnallite and mixed bischofite-carnallite-halite samples;
- Average length = 80 mm, diameter = 35 mm;
- Jacketed and undried samples with deliquescence conditions;
- Triaxial laboratory tests at real *in-situ* conditions P_c = 40 MPa, T = 70 °C;
- Strain rate stepping, with stress relaxation (**eq. 1**) after selected constant deformation steps.

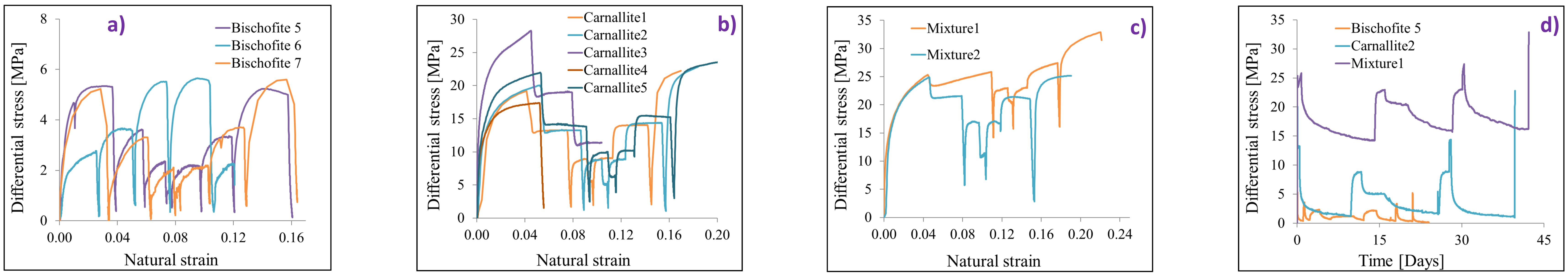

*Figure 1 . Mechanical data; differential stress vs. natural/true strain for bischofite **(a)**, carnallite **(b)** and mixtures **(c)**, and differential stress vs. time for three representative samples **(d)***

3. Results:

- Strength of all salts is strain rate sensitive;
- Carnallite is 4-5 times stronger than bischofite, while the mixtures were stronger than carnallite (**Fig. 1**);
- Steady state stress not reached for mixtures;
- Strength of mixtures is directly related to the halite content (**Fig. 2**)

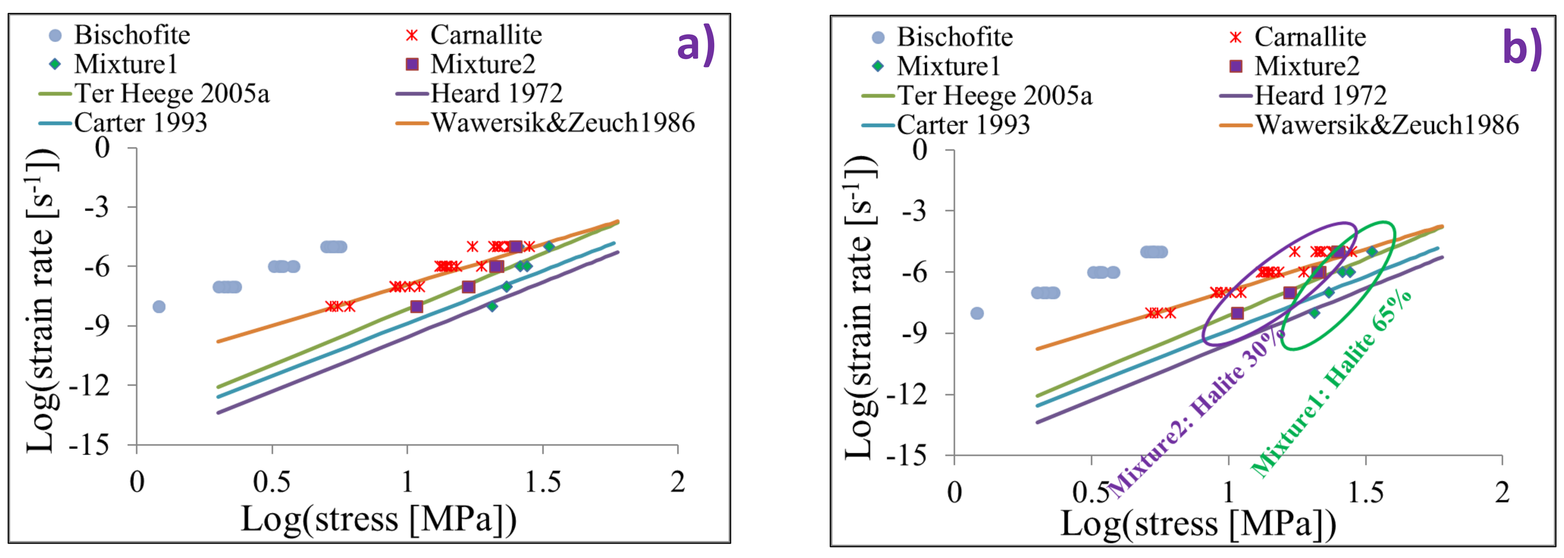

*Figure 2 **a)** Projected curves for flow laws of wet halite from previous studies, **b)** steady state values comparison of bischofite, carnallite and mixture (with halite% composition)*

4. Stress relaxation behaviour:

- During stress relaxation (hardly any straining), the stress exponent n in a power law (**eq. 2**) gradually changed from $n > 5$ at 10^{-5} s^{-1} to n~1 at 10^{-9} s^{-1} (**Fig. 3**);
- This suggests a gradual change in mechanism with decreasing strain rate, from grain size insensitive (GSI, **eq. 2**) dislocation creep to grain size sensitive (GSS, **eq. 3**) pressure solution creep;
- The relaxation curves do not pass through the steady state values: suggests differences in microstructure evolution.

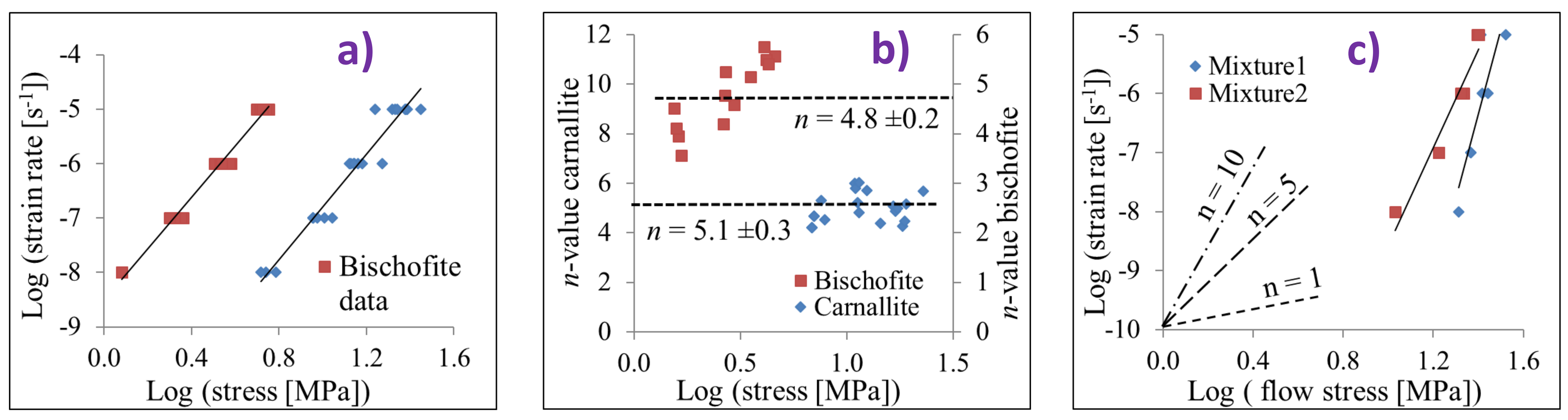

*Figure 4. **a)** Steady state stress values of bischofite and carnallite against strain rate in log space, **b)** n-value at different steady state stress values in bischofite and carnallite, **c)** n-value at different steady state stress values in mixture*

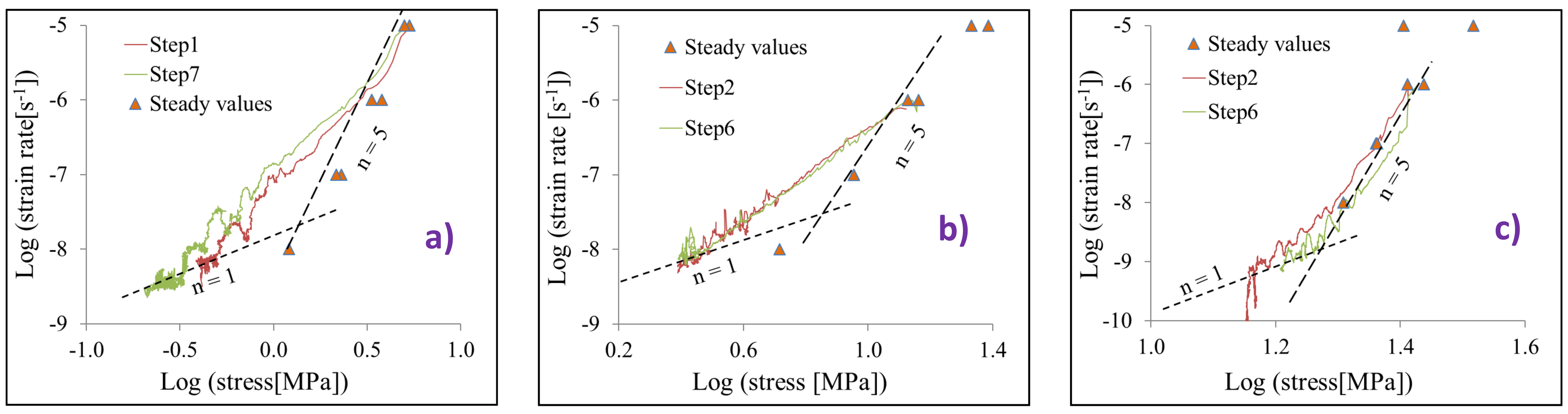

*Figure 3. Stress relaxation curves **a)** bischofite5, $\dot{\varepsilon} = 10^{-5}$ s^{-1}, **b)** carnallite2 $\dot{\varepsilon} = 10^{-6}$ s^{-1}, **c)** mixture1 $\dot{\varepsilon} = 10^{-6}$ s^{-1}*

5. Important equations:

Plastic strain rate ($\dot{\varepsilon}_{plastic}$) during stress relaxation

- $\dot{\varepsilon}_{plastic} = \dot{\varepsilon}_{total} - \frac{1}{E_{sample}}(\dot{\sigma}_{sample}) - \frac{1}{L_t} S\left(\frac{\partial F}{\partial t}\right)$ (1)
- Grain size insensitive creep(GSI): $\dot{\varepsilon} = A\sigma^n$ (2)
- Grain size sensitive creep (GSS $\dot{\varepsilon} = B\sigma^n d^{-p}$ (3)
- Composite equation (GSI+GSS): $\dot{\varepsilon} = A^*\sigma^n + B^*\sigma d^{-p}$ (4)

***Composite Flow law** (σ in MPa, d in mm):*

- **For bischofite:** $\dot{\varepsilon} = 1.1 \times 10^{-9}\sigma^{5.4} + 3.94 \times 10^{-8}\sigma d^{-0.8}$ (5)
- **For carnallite:** $\dot{\varepsilon} = 3.70 \times 10^{-13}\sigma^{5.3} + 1.01 \times 10^{-8}\sigma d^{-1}$ (6)

6. Interpretations and Conclusions:

- The difference between the stress relaxation and the constant strain rate parts of the tests can be explained by assuming constant microstructure during the former and grain size evolution (by dynamic recrystallization) during the latter (**Figs. 3-5**);
- Composite creep laws combining GSI creep and GSS creep, holding at 70 °C, were established for bischofite and carnallite (**eqs. 5 and 6**) based on the GSI flow laws (@ constant strain rate steps) and the change to GSS (@ stress relaxation). **Figs. 6.**
- The composite creep laws form the better descriptions in cases that effective microstructural modification cannot be assumed, as for example during transient creep in the walls of salt caverns.

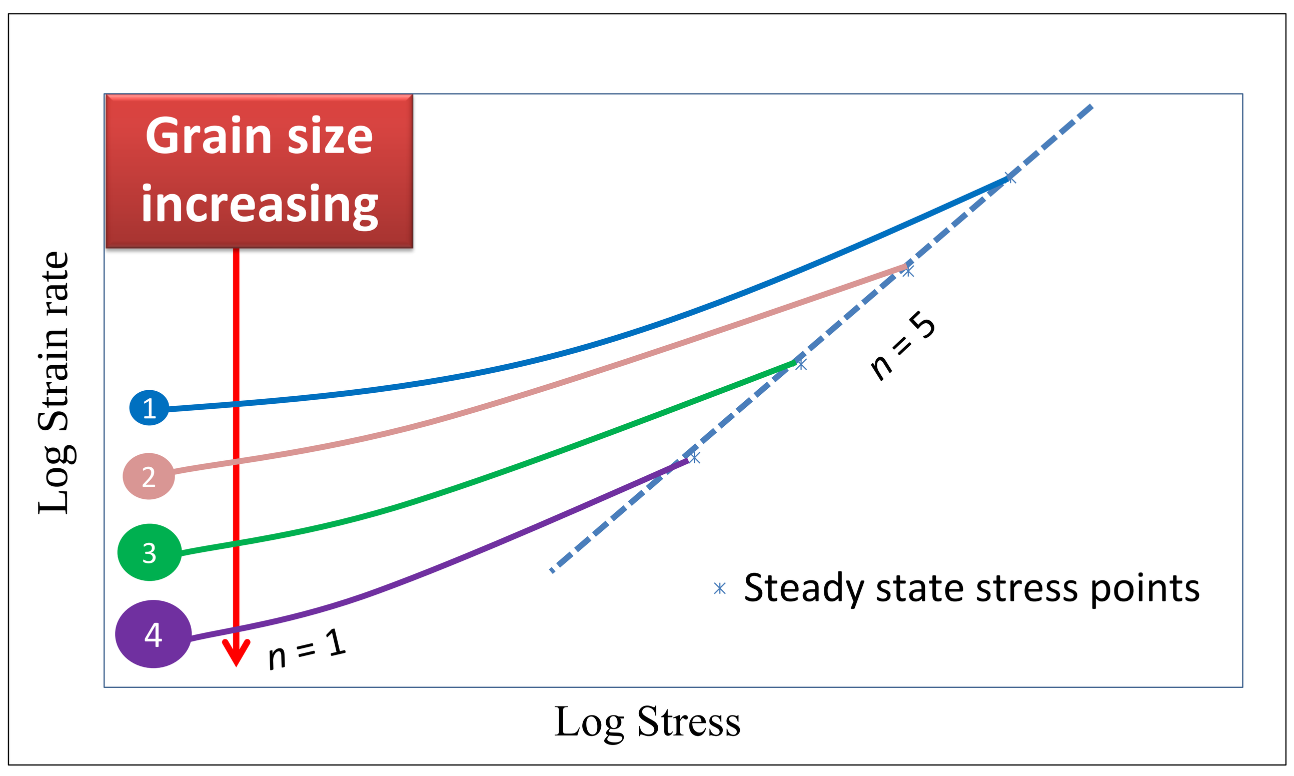

Fig. 5 Schematic diagram: Changing n-value during relaxation

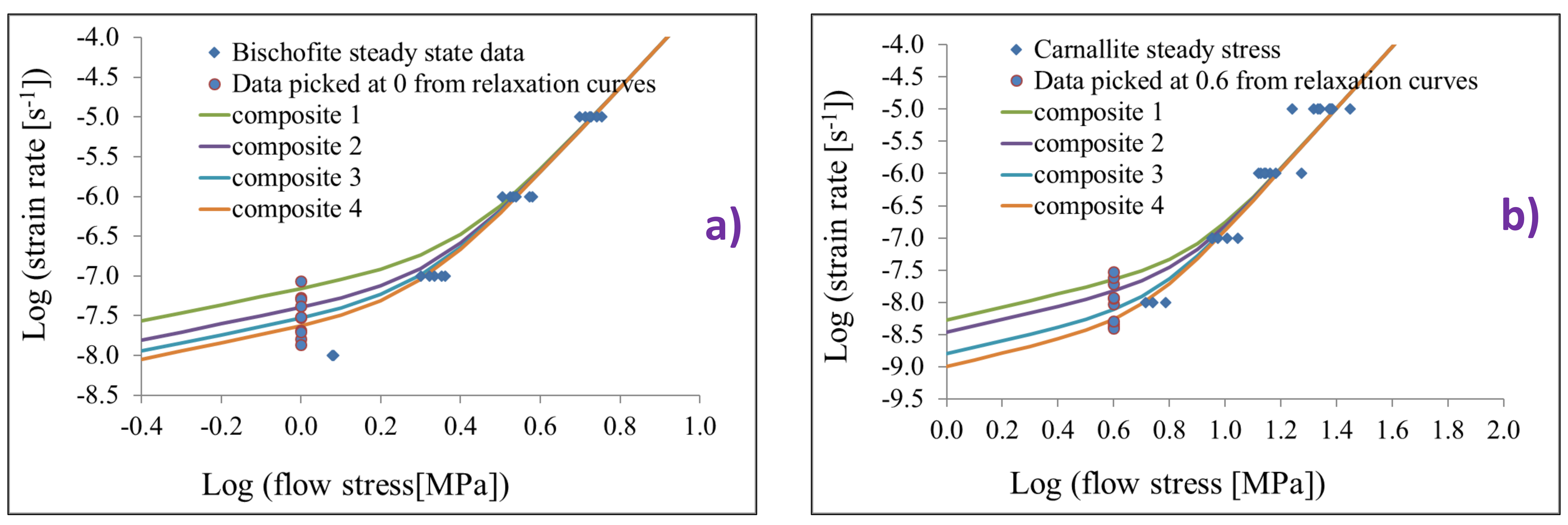

*Figure 6. Trends resulting from the composite GSI + GSS flow law covering the steady state behaviour (diamond data points) as well as the gradual decrease in n-value during relaxation (from n ~ 5 to n ~ 1 when going towards low stress and strain rate), **a)** bischofite, **b)** carnallite*

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