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DETERMINATION OF MECHANICAL PARAMETERS OF SALTS IN THE CYCLIC LOAD PROCESSES CORRESPONDING TO THE OPERATION OF A STORAGE CAVERN FOR HYDROGEN

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Abstract: The paper describes the results of laboratory tests of the strength of salt samples made as part of the HESTOR project in order to determine the mechanical parameters of salt. The tests were carried out using an Autolab 2000 apparatus which allows to simulate any load cycles. The tests were made by simulating the operation of the hydrogen storage cavern. In order to observe the differences in salt behavior depending on the sample medium being stored during the test, gases were supplied: nitrogen, as an analogue of natural gas, and helium as a hydrogen analogue.

Keywords: salt, hydrogen, storage cavern, energy storage in the hydrogen

1. Introduction

Strength tests to determine the mechanical parameters of salt were made using an Autolab 2000. The tests were carried out for both the “pure” salt sample and the samples to which nitrogen was supplied, as an analogue of natural gas and helium as a hydrogen analogue [1].

Autolab 2000 is a specialist apparatus for testing the strength and petrophysical properties of rock samples, enabling the implementation of any load scenario in a triaxial stress system. The apparatus allows the examination of samples with a maximum core diameter of 102 mm (4 inches) and a length of up to 177.8 mm (7 inches). The maximum axial load on the tested core is up to 1890 kN, with the maximum sealing pressure up to 140 MPa. The maximum pore pressure in the core under test is also 140 MPa [2].

Measurements of mechanical properties of salt samples cut from selected locations consisted of placing a sample prepared in the form of a cylinder with a diameter of 1.5 inch and a height of 3–3.5 inch, so that the slenderness 2 (the height of the sample to its diameter) was preserved, in a special rubber flange, which from the bottom and top is closed with steel pivots. Special rings are mounted on them to place the LVDT sensors around the sample, and then place it in the apparatus. The prepared salt sample is placed on the apparatus table (presented in Fig. 1) to which LVDT sensors are connected, which makes it possible to read the sensors in real-time using specialized software [2].

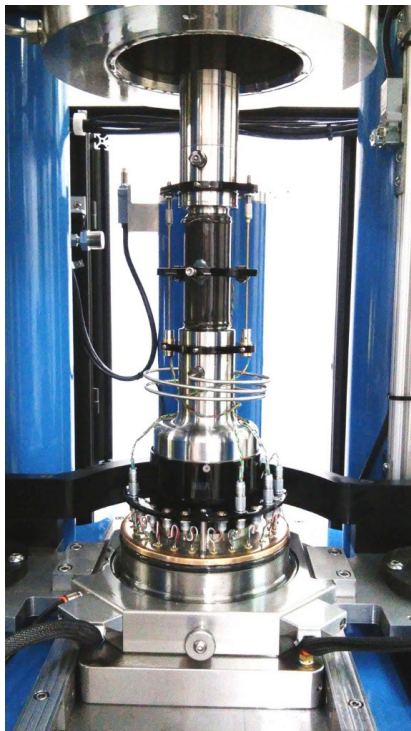


Fig. 1. The prepared salt sample during placement in the Autolab 2000 measuring cylinder

The sample placed on the table is closed into the pressure chamber of the apparatus. The chamber is filled with oil which creates peripheral pressure (seal pressure) around the sample. Then, by controlling the movable piston, pressure is applied to the salt sample previously described by distances, whereby the axial stresses in the test sample are induced. On the basis of readings from linear and peripheral strain sensors, the computer software processes the obtained results. Thanks to this it is possible to determine mechanical parameters during a given measurement cycle [2].

2. Laboratory tests of mechanical parameters of samples of salt cores as an elastic-plastic-viscous medium in cyclic load processes

To perform laboratory tests of mechanical properties, samples were selected from three different locations of the Polkowice-Sieroszowice Mine, representing various crystallographic salt structures [3–6]. Samples were made in the form of a cylinder with a diameter of 38.1 mm (1.5 inch) and a height of approx. 86 mm (3.4 inch), which were adjusted to the requirements of the Autolab 2000 apparatus [2].

The following sample description scheme was adopted: PS7, US2, SG2 indicates the location from which the given sample originated. Roman number I, II, III indicate the number of the sample that has been tested. The Arabic number 1, 2, 3 indicates the next test number for the given sample. Investigations of mechanical properties of salts from selected locations were conducted in short-term load cycles using the same scenario for selected samples. In the case of samples where no gas was supplied, a confining pressure was set to 10 MPa.

For samples where gas (nitrogen or helium) was supplied at pressure 5 MPa, the confining pressure was increased to 15 MPa to maintain the same test conditions as for the “clean” samples. For all samples, the vertical pressure was set at the beginning of the test so that the piston loading the sample adhered to it but did not load it – without any initial deformation.

The test scenario was as follows: after setting the confining pressure (and the saturation pressure), the salt sample was left until the deformations observed in the graph (live data) showing that the sensors remained unchanged. Depending on the sample, it took 0.5–2.5 h. In the next step, the axial load of the sample was set by

changing the pressure exerted by the piston loading the sample (the pressure exerted by the piston). The amplitude of this load was 4 MPa. After reaching 4 MPa, the load was removed. For the tests, a load speed of 0.006 MPa/s was adopted, i.e. the loading time was 666.7 s and the same unloading time. In order to compare the impact of the speed of a given load on the obtained results, during the tests it was decided that for one load of the same sample, a 10× speed higher, i.e. 0.06 MPa/s, would be used. The loading time of the sample in this case was therefore 66.7 s and the same unloading time. Each of the tested samples was loaded 3 times, with a 48–72 hour period between successive loads. At that time, the sample was removed from the apparatus – unloaded.

It should be emphasized that the adopted research scenarios assumed non-destructive testing, as the samples used were to be comparably tested to determine permeability. Therefore, the tests were performed in the lower range of loads used for salt, which translates into Young's modules being obtained which belong to this load range. At higher loads and relieves, the values obtained can be greater, even five times as much.

The tests were performed using LVDT (Linear Variable Displacement Transducer Test) sensors. These sensors operate on the principle of an induction coil producing 0–10 V voltage. Through the software, the signal from the sensors is converted into a displacement value. Each time the prepared sample was placed on the Autolab 2000 table, the LVDT sensors were calibrated by placing the sensor core as close as possible to the center of the coil. The calibration was made by setting a voltage close to 1.5 V for axial sensors and a value close to 5 V for a radial sensor, so that the range of sensor readings was as large and as accurate as possible. However, the high sensitivity of the sensors means that even after the calibration is carried out directly on the sample, there is the phenomenon of “initial deformation” of the sample that can also be caused by the reaction of the sensors to apply the initial axial pressure to the sample (touching the sample by the loading piston).

During the test, the readings of deformation values of samples from 2 linear strain sensors (LVDT1, LVDT2) and a radial strain sensor (LVDT3) were carried out. The examples of data from LVDT sensors obtained during testing are shown in the diagrams presented in Figures 3, 5 and 7. With the use of LVDT sensors, the computer software allows the reading of axial deformations (change of sample height) and radial (change of sample diameter) as the ratio of the sample height/diameter (before the start of the test) to the current value of this parameter.

$$\varepsilon = \frac{\Delta l}{l} \quad (1)$$

The accuracy of LVDT sensors allowed us to obtain results with an accuracy of 10^{-16} , hence the unit in which the presented results in the graphs is “ $1m\varepsilon$ ” (as described in the Autolab 2000 charts), i.e. 1‰. On the basis of readings from sensors inside the apparatus allowing control over the pressure loading the sample, the software automatically determines the values of the Young's modulus and Poisson's coefficient for the tested sample. In combination with the high sensitivity of LVDT sensors, the Autolab 2000 apparatus allows the values of these coefficients to be obtained with an accuracy of 10^{-9} , however for the purpose of determining these parameters for salt, such high accuracy is not required, hence the accuracy of these 10^{-3} values in the results presented in Tables 1–3 [2].

3. Test results

Sample PS7

Sample PS7 is presented in Figure 2. This is a very clean sample with large salt crystals, a white-colored crystal. The halite crystals have sizes up to 3 cm. Their outlines are xenomorphic, uneven and show no elongation. Occasionally, pollutants appear in the form of small anhydrite streaks distributed along the boundaries of the halite crystals [5].

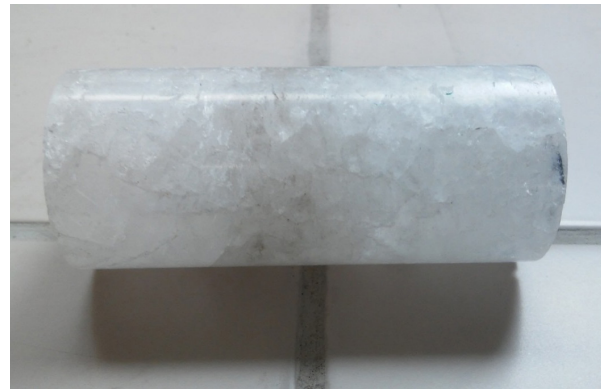


Fig. 2. Sample PS7 – halite crystals have sizes up to 3 cm. Their outlines are xenomorphic and showing no elongation

The example of deformation diagram obtained during the tests of samples PS7 are presented on Figure 3. The diagram include direct readings from sensors LVDT1, LVDT2 (axial deformations – decrease of the sample height), LVDT3 (radial deformations – reduction of the sample diameter). When comparing individual diagrams, it can be noticed that axial deformations

during the tests for samples from this location did not exceed 0.3‰. After unloading the sample, its total strain did not exceed 0.15‰. However, radial deformations

only occurred when the maximum load of the sample was reached. After relieving the samples, they returned to their original level.

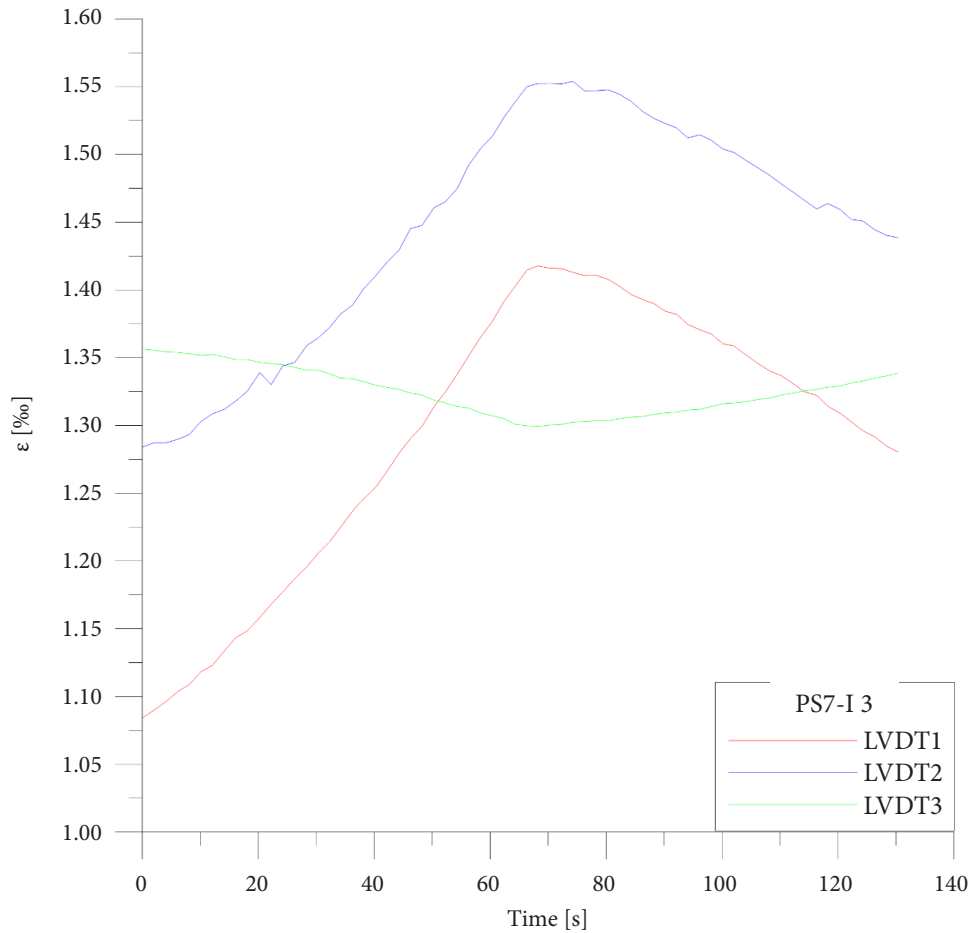


Fig. 3. The reading diagram from sensors LVDT 1, 2, 3 obtained during the test for sample PS7-I 3

Table 1. Results obtained during testing the PS7 sample

Sample	Sample diameter [mm]	Sample height [mm]	ν	E [GPa]	Temperature [°C]	Pore pressure [MPa]	Confining pressure [MPa]	Gas in the pores	Loading speed [MPa/s]
Ps7-I 1	38.15	86.85	0.205	13.790	16.809	0.00	10.175	none	0.006
Ps7-I 2	38.15	86.85	0.133	8.659	16.311	0.00	10.153	none	0.006
Ps7-I 3	38.15	86.85	0.181	9.605	16.113	0.00	10.153	none	0.06
Ps7-II 1	38.10	86.80	0.225	26.989	17.145	5.00	15.186	N ₂	0.06
Ps7-II 2	38.10	86.80	0.229	26.824	16.347	5.00	15.181	N ₂	0.06
Ps7-II 3	38.10	86.80	0.207	20.866	16.604	5.00	15.173	N ₂	0.006
Ps7-III 1	38.05	86.65	0.230	21.162	16.563	5.00	15.185	He	0.06
Ps7-III 2	38.05	86.65	0.235	15.793	16.873	5.00	15.191	He	0.006
Ps7-III 3	38.05	86.65	0.248	21.197	16.2996	5.00	15.174	He	0.06

In samples from the PS7 location (Tab. 1), Poisson's values obtained during tests of "clean" and gas-saturated samples fluctuated in the range of 0.13–0.24, while the Young's modulus for samples without saturation varies between 8.65–13.78 GPa. For samples "with helium": 15.7–21.19 GPa, and the highest value was obtained for samples which were saturated with nitrogen: 20.86–26.98 GPa.

On the basis of the obtained results, it can be concluded that the gas saturation has no effect on the change of the Poisson coefficient, while the salt subjected to gas saturation shows an increase in the Young's modulus. The spread of the obtained results, both for nitrogen and helium, does not allow for unambiguous confirmation of whether the type of gas used affects the differences in the Young's modulus obtained.

Sample US2

This sample is presented in Figure 4. It is a light gray, medium and coarse crystalline salt with halite crystals up to 1 cm. A rock with a random texture, in places the halite crystals have a slight elongation. After a few hits the sample becomes crumbly, "sugary" [7].



Fig. 4. Sample from the US2 location – in the picture visible light gray, medium-crystalline salt construction

The example graph of deformations of samples from the US2 location during the tests are presented in Figure 5. The deformation scale for US2 samples does not exceed 0.3‰ relative to the state at the beginning of the load cycle.

It should be noted that the LDVT1, LDVT2 diagrams for US2 and 1, 2, 3 trials show only a very low amplitude, which is due to the large y-axis range. The large "initial deformation" of the US2-I sample is probably caused by inaccurate calibration of the sensors before the start of the test, hence the high "initial strain" value for this sample [8].

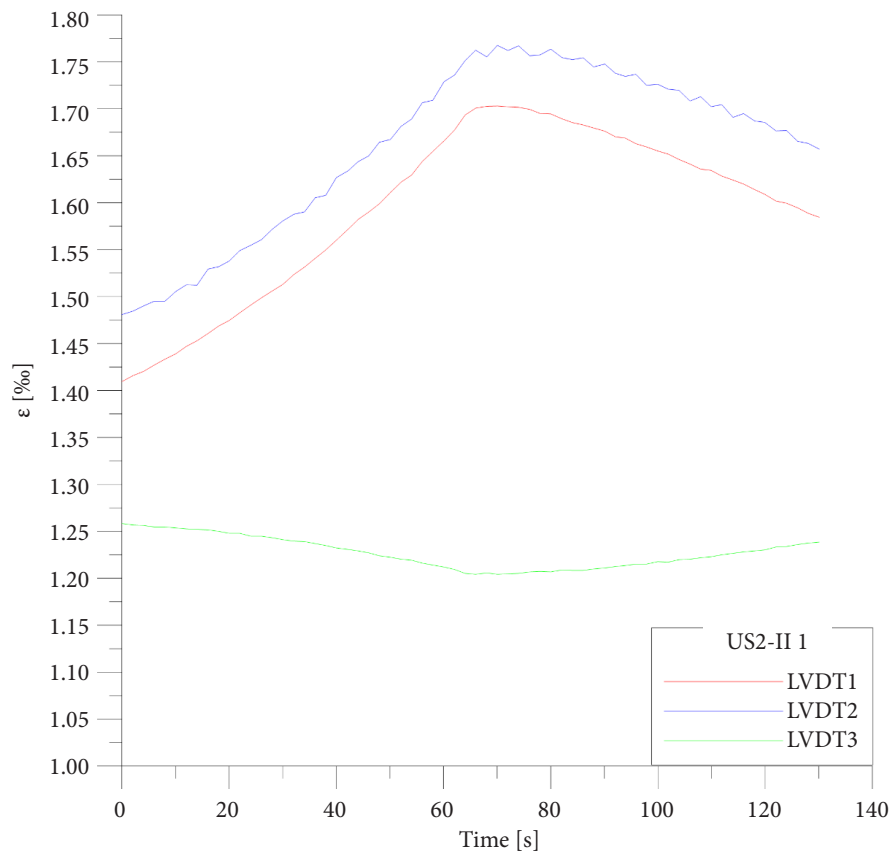


Fig. 5. The reading diagram from sensors LVDT 1, 2, 3 obtained during the test for sample US2-II 1

Table 2. Results obtained during testing the US2 sample

Sample	Sample diameter [mm]	Sample height [mm]	ν	E [GPa]	Temperature [°C]	Pore pressure [MPa]	Confining pressure [MPa]	Gas in the pores	Loading speed [MPa/s]
US2-I 1	38.05	86.00	0.303	6.361	17.189	5.00	12.226	N ₂	0.06
US2-I 2	38.05	86.00	0.304	4.137	17.550	5.00	15.254	N ₂	0.006
US2-I 3	38.05	86.00	0.323	5.792	17.431	5.00	15.272	N ₂	0.006
US2-II 1	38.50	88.25	0.181	9.624	16.921	5.00	15.254	He	0.06
US2-II 2	38.50	88.25	0.250	12.661	17.000	5.00	15.222	He	0.06
US2-II 3	38.50	88.25	0.208	7.205	16.436	5.00	15.230	He	0.006
US2-III 1	38.15	85.75	0.268	11.776	16.851	0.00	10.163	none	0.06
US2-III 2	38.15	85.75	0.298	8.108	16.482	0.00	10.167	none	0.006
US2-III 3	38.15	85.75	0.310	14.494	16.661	0.00	10.177	none	0.006

For the “clean” samples from US2 location (Tab. 2), Poisson’s ratios of 0.26–0.31 were obtained, for samples saturated with nitrogen: 0.30–0.32, while for helium-saturated samples, the values of this coefficient varied within 0.18–0.25.

The Young’s modulus values were respectively: 8.1–14.5 GPa for “pure” samples, 4.13–6.36 GPa for samples saturated with nitrogen, 7.2–12.66 GPa for samples saturated with helium. On the basis of the obtained results, in the case of Poisson’s coefficient one can notice a slight decrease in its value with samples with helium. However, on the basis of the value of this coefficient obtained for the “pure” and saturated with nitrogen, the effect of gas saturation on the change of the Poisson coefficient cannot be confirmed.

In the case of Young’s modulus, the lowest values of this parameter were obtained for samples saturated with nitrogen, which may indicate a weakening of the geomechanical properties of salts under the influence of gas. However, already the comparison of the values obtained for “pure” and helium samples, the differences in the values of this parameter are not so clear and do not allow unambiguous confirmation of this conclusion.

Sample SG2

A sample of white or light gray medium crystals with a random texture (Fig. 6). The sample is similar to sample US2, and after a few hits it becomes scattered. The halite crystals have sizes of about 3 to 7 mm [7].

A sample deformation diagram obtained during the tests of samples from the SG2 location is present-

ed in Figure 7. When comparing individual diagrams, it can be noticed that the scale of axial deformations during the tests, for samples from this location did not exceed 0.3‰, while radial deformations for SG2 samples were practically non-existent.

For the “clean” samples from SG2 location (Tab. 3), Poisson’s values of 0.05–0.10 were obtained, for samples saturated with nitrogen: 0.13–0.21, while for samples saturated with helium, values of this coefficient were obtained: 0.13–0.22. In the case of Young’s modulus, the values were respectively: 13.05–14.06 GPa for “pure” samples, 19.98–25.12 GPa for nitrogen saturated samples and 22.23–30.62 GPa for helium saturated samples.



Fig. 6. Salt sample SG2. The picture shows a disorderly, medium-crystalline salt structure

In the case of samples from this location, a large difference in the obtained parameter values is noticeable compared to the tested salt samples from other locations. This may be due to the significant differences

in the mechanical properties of the salt from this location, which can be influenced by inclusions inside all of the salt samples from this location. In the case of samples saturated with nitrogen and helium, there is also

a clear increase in the value of the tested parameters, however, based on the conducted tests it is impossible to clearly indicate whether the type of gas used affects the values obtained.

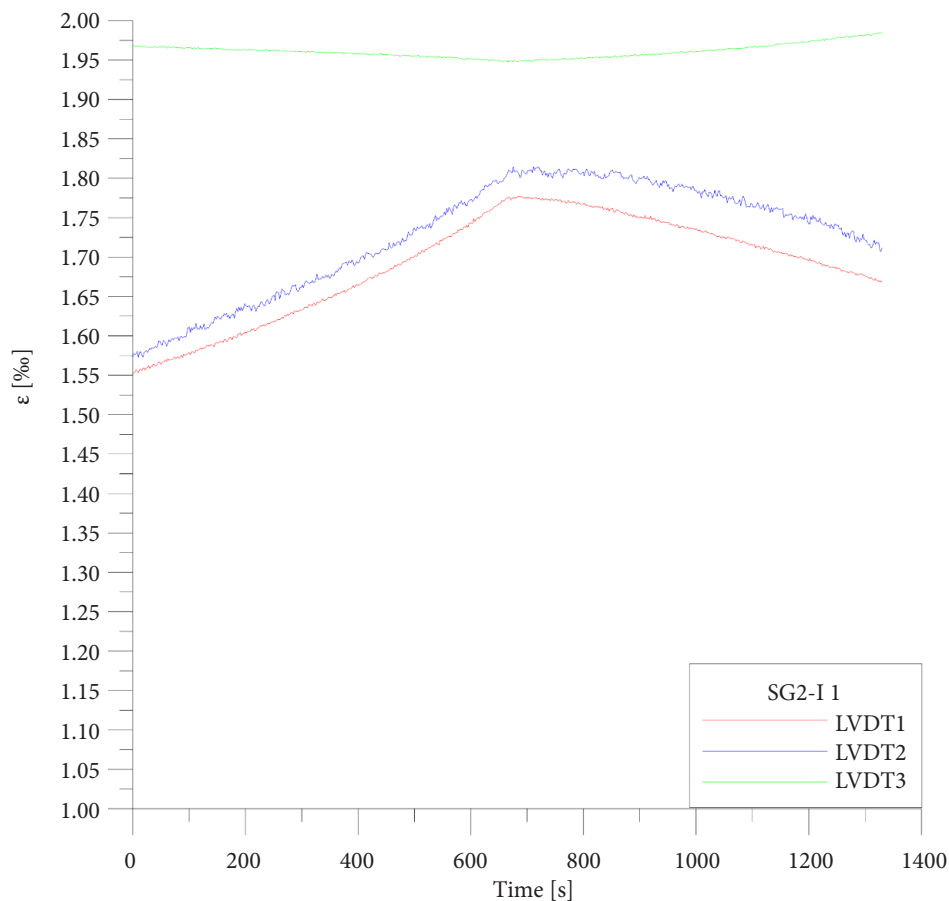


Fig. 7. The reading diagram from sensors LVDT 1, 2, 3 obtained during the test for sample SG2-I 1

Table 3. Results obtained during testing the SG2 sample

Sample	Sample diameter [mm]	Sample height [mm]	ν	E [GPa]	Temperature [°C]	Pore pressure [MPa]	Confining pressure [MPa]	Gas in the pores	Loading speed [MPa/s]
SG2-I 1	38.05	86.02	0.056	13.377	17.278	0.00	10.205	none	0.006
SG2-I 2	38.05	86.02	0.105	14.060	17.142	0.00	10.200	none	0.06
SG2-I 3	38.05	86.02	0.096	13.058	16.101	0.00	10.145	none	0.06
SG2-II 1	38.15	85.8	0.132	23.176	15.667	5.00	15.188	N ₂	0.006
SG2-II 2	38.15	85.8	0.163	19.984	17.689	5.00	15.251	N ₂	0.006
SG2-II 3	38.15	85.8	0.215	25.120	13.584	5.00	15.101	N ₂	0.06
SG2-III 1	38.17	86.91	0.156	30.615	17.149	5.00	15.204	He	0.006
SG2-III 2	38.17	86.91	0.134	22.231	16.175	5.00	15.195	He	0.006
SG2-III 3	38.17	86.91	0.218	26.785	12.933	5.00	15.121	He	0.06

4. Conclusions

The results of mechanical properties tests for samples from the US2 location confirm the average values of the Young's Module obtained so far in uniaxial mechanical tests ($E \approx 8$ GPa). However, for samples from the location of SG2, PS7, higher values of the Young's modulus were obtained, which may be due to the overly large number of crystals in relation to the total size of the tested sample or/also a larger number of anhydrite inclusions inside the salt samples.

During the tests, the long relaxation time of the salt was indirectly confirmed – for the tested samples at the repetition of the load cycle even after 2–3 days higher values of the Young's modulus were obtained. It may also be due to stress reinforcement of the sample. On the other hand, the Poisson ratio was similar for each specific sample.

During the determination of the Poisson ratio, similar values were obtained for the majority of samples ranging from 0.1 to 0.3. This may indicate that the construction of salt crystallographic salt does not directly affect the value of this coefficient [9].

During the tests carried out with the use of two sample loading speeds (0.006 MPa/s and 0.06 MPa/s), no unambiguous effect of the speed of the given load on the obtained values of mechanical parameters of the tested samples was observed.

The results of laboratory tests of mechanical properties with nitrogen and helium saturation did not show significant differences in the mechanical parameters tested. It confirms that it is possible to store hydrogen in salt caverns and the storage will not be significantly different from the case of natural gas storage [10].

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