

## COMPARISON OF UNCONFINED COMPRESSIVE STRENGTHS AND ACOUSTIC EMISSIONS OF ESTONIAN OIL SHALE AND BRITTLE ROCKS

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**Abstract.** *The purpose of this study was to confirm the existence of acoustic emission (AE) Kaiser effect (KE) in Estonian oil shale (OS), and compare the respective data obtained about working mine pillars. The KE of acoustic emission, a phenomenon with a potential for in-situ stress estimation, can be used for quantifying the damage levels of pillars, and possibly even to measure the state of stress within a pillar. The main role of measurements is to confirm estimated stresses, as the estimation is quite simple in regions of sedimentary rocks. The performed tests showed that the Kaiser effect did exist in oil shale material, at the same time, the low material strength also lowered the feasible stress limit for KE-based stress measurements. Tests were made with inspection of the formula for changes of long-term rock durability in the Estonian oil shale formation. On the basis of the obtained results, the AE method can be used to estimate the long-term rock durability for pillars in conditions of Estonian oil shale mines.*

**Keywords:** *compressive strength, acoustic emission, Kaiser effect, Estonian oil shale, brittle rocks.*

### 1. Introduction

The oil shale (OS) layers occur between the limestone layers in the Kukruse Middle Ordovician Regional Stage, Estonia. The oil shale is a stratified sedimentary rock which is rich in organic matter (15–46 % kerogen, 26–57 % carbonates, 18–42 % clastic materials). The commercial oil shale bed consists of six OS layers, which are specified from bottom to top by the indexes A, A1, B, C, D, E, F1, and four interbedded limestone layers designated as A1/B, B/C, C/D, D/E. Stratigraphic column indexes (oil shale and limestone) and compressive strengths of layers are presented in Table 1. The mechanical characteristics of oil shale and limestone layers are quite different. The compressive strength of oil shale is 16–40 MPa and that of

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limestone, 60–85 MPa (Table 1). The volume density of the rocks varies between 1500 and 1900 kg/m<sup>3</sup> and from 2200 to 2600 kg/m<sup>3</sup>, respectively, while their strength increases in the southward direction [1, 2].

**Table 1. Stratigraphic column indexes and compressive strength of the oil shale bed**

Layer index	Thickness, m	Height from layer A, m	Compressive strength, MPa
F	0.42	2.8	18
E	0.61	2.38	18
D/E	0.07	1.77	67
D	0.06	1.7	29
C/D	0.29	1.64	82
C	0.41	1.35	26
B/C	0.12	0.94	75
B	0.38	0.82	40
A <sub>1</sub> /B	0.18	0.44	65
A <sub>1</sub>	0.14	0.26	26
A	0.12	0.12	32

### 1.1. Long-term strength of the rocks

Rooms and intervening pillars are the constructive elements of room-and-pillar mining systems. Many technical and economic parameters of production, including loss of OS reserves, depend on the correct choice of the sizes of these elements.

Rock strength data are of key importance for the choice of the sizes of constructive elements used in room-and-pillar mining. Without taking into account the rheological properties of rocks, in particular the character of the change in their long-term durability, the calculation of the sizes of rooms and pillars for a certain term is impossible [3].

The character of changes in the durability of the oil shale bed and roof limestone strata is described with sufficient accuracy by the following empirical formula devised by The State Research Institute of Mining Geomechanics and Mine Surveying (VNIMI), St. Petersburg [1–4]:

$$k_t = \alpha + \beta \left( \frac{1}{1+t} \right)^m, \quad (1)$$

where  $k_t$  is the factor of the change in durability of rocks in time, equal to the ratio of current durability  $R_t$  to initial durability  $R_0$  [3, 4];  $\alpha$ ,  $\beta$  and  $m$  are empirical factors reflecting properties of rocks ( $\alpha = 0.44$ ;  $\beta = 0.56$ ;  $m = 0.6$ );  $t$  is the service life of pillars or rooms, in months.

When the service life of pillars  $t$  approaches infinity ( $t \rightarrow \infty$ ), the limit value of factor  $k_t$  equals 0.44. The dependence of rock durability on time established by experimental way is shown in Figure 1.

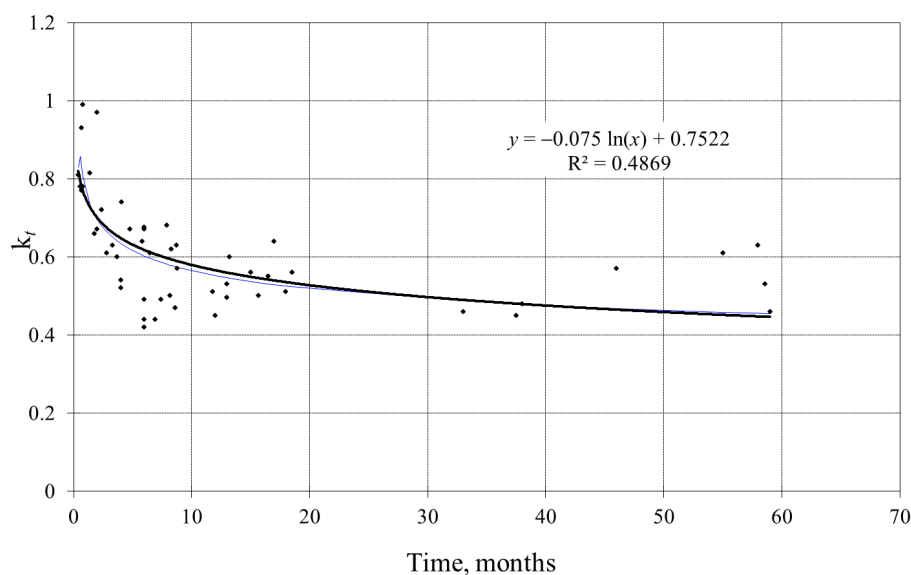


Fig. 1. Dependence of rock durability on time.

The above equations are valid in case of vertical loading as oil shale has a clear micro-layered structure because of a parallel orientation of the flattened organic agglomeration and oblong carbonate oddments of organisms. This may cause some strength anisotropy, which means that in the parallel direction the micro-layered part may collapse easier than in the perpendicular direction [2, 4].

Proceeding from Equation (1), the durability of Ordovician rocks in conditions of the Baltic Oil Shale Basin can be established using Equation (2):

$$R_t = k_t R_0, \quad (2)$$

where  $R_t$  is the current durability of rock,  $k_t$  is the factor of the change in durability of rocks in time,  $R_0$  is the initial durability of rocks.

The purpose of this work was to confirm the existence of acoustic emission (AE) Kaiser effect (KE) [5] in Estonian oil shale, and to compare the respective data obtained about working mine pillars (Fig. 1). KE can be used for quantifying the damage levels of pillars, and possibly even to measure the state of stress within a pillar. The main role of measurements would be confirmation of estimated stresses, as the estimation is quite simple in regions of sedimentary rocks. Plus, the simple geometry of the room and pillar mining method facilitates the stress estimation of pillars even further.

## 2. Samples and specimen preparation

Oil shale samples were taken from three different pillars of the Estonia mine, two from the active mining block #3104 where emulsion explosives were used (sampling pillars #2 and #3, Table 2) and one from the mining block #3106 where cartridge explosives were used (sampling pillar #1). The geological conditions were normal and sampling depth was 65 m (layer B). The sampling depth from the pillars wall was about 1 m. The lifetime of pillar #3 at the sampling moment was 23 days and its cross sectional area was about 50 m<sup>2</sup>. Pillar #1 had a lifetime of 26 months and area of 42 m<sup>2</sup>. Pillar #2 was 20 months old with a cross sectional area of 48 m<sup>2</sup>. The blocks were of irregular shape. Oil shale often contains limestone inclusions, and one block sample had to be rejected because of this.

A total of 10 cores, six of 21 and four of 32 mm diameter, were core-drilled from the bulk samples. The cores were first trimmed to a length/diameter ratio of 2.5, and the trimmed cores had their ends ground parallel with a tolerance of 0.01 mm, according to the recommendations given by the International Society for Rock Mechanics (ISRM) in the method for uniaxial compression testing [6]. Sampling information has been summarized in Table 2.

**Table 2. Sampling information**

Sample ID	Sampling pillar	Diameter, mm	Orientation (in respect to foliation)	Notes
Ref. 2	1	21	Parallel	
Ref. 3	1	21	Perpendicular	
Ref. 4	1	21	Parallel	Failed during test
Ref. 5	2	21	Perpendicular	Broken, not tested
Ref. 6	2	21	Parallel	
Ref. 9	3	32	Perpendicular	Tested to failure
Ref. 10	3	32	Perpendicular	
Ref. 11	3	32	Perpendicular	Tested to failure
Ref. 12	3	32	Perpendicular	

## 3. Testing equipment and methods

The testing was performed using a servo-hydraulic MTS-815 material testing system. A separate acoustic emission recording system was used for searching the Kaiser effect. This phenomenon is a property of acoustic emission that appears during the cyclic loading of a material.

Acoustic emission is defined by the American Society For Testing And Materials (ASTM) as “the class of phenomena whereby transient elastic waves are generated by the rapid release of energy by a localized source or sources within a material, or the elastic waves so generated.” AE is generated in practically all solid materials which are loaded by external or

internal forces, which in turn cause micro-scale deformations. In rock materials these are mostly associated with closure and propagation of mineral grain size [7].

Acoustic emission is typically monitored with piezoelectric transducers that convert the displacement vibrations into electric pulses. These pulses are then pre-amplified before they are passed into the recording unit.

Kaiser effect is defined by ASTM as “the absence of detectable acoustic emission until previously applied stress levels are exceeded”. It is connected to an ability of material to “remember” the previously applied stress, as it is one of the “memory” indicators. Other two indicators are the gradient change of inelastic strain and a characteristic change in the propagation velocity of elastic waves. All three indicators display a probable link between applied stress and mechanical behavior of micro-cracks [7].

In reality KE is seldom perfect, and a parameter called Felicity Ratio (FR) is used to measure the quality of KE. It is simply calculated as the ratio of the known previous stress level to the turning point stress level of the graph and is expressed as percentage.

This memory effect of rocks has one highly attractive application: it can be used to estimate the in-situ state of stress in bedrock. The estimation method uses oriented (core) samples taken from the estimation area, which are re-sampled in six independent directions. The memorized stress level is then sought out using the Kaiser effect and the results are inverted to obtain the full 3D stress tensor.

This stress memory has been proven to exist in brittle rocks and some types of plastic rocks as well. In Finland, KE has previously been utilized for estimation of rock in-situ stresses at Olkiluoto [8]. Worldwide, the method has had most use in Australia and China. No previous applications of KE to oil shale stress estimation are known, so this work attempts to fill the gap.

The Mistras-2001 acoustic emission system used in this work is a computerized unit that processes and records the incoming pulses. The system filters the arriving signals, performs feature extraction and digitizes the waveform. Typical extracted features are, e.g., amplitude, duration and the amount of counts (pulses) in a single event (Fig. 2). An event is basically a train of pulses at different frequencies and amplitudes. The saved data always contains the extracted features; the digitized waveform can also be included.

#### 4. Methods

The specimens were tested for the artificial Kaiser effect according to the test sequence which was used by Hunt et al. [9] and named the load-reload-reload (LRR) test. In the researchers' work, the specimens were preloaded with a loading rate of 4.1 MPa/min to a suitable stress level, the load was held for one hour after which the specimens were rapidly unloaded. The

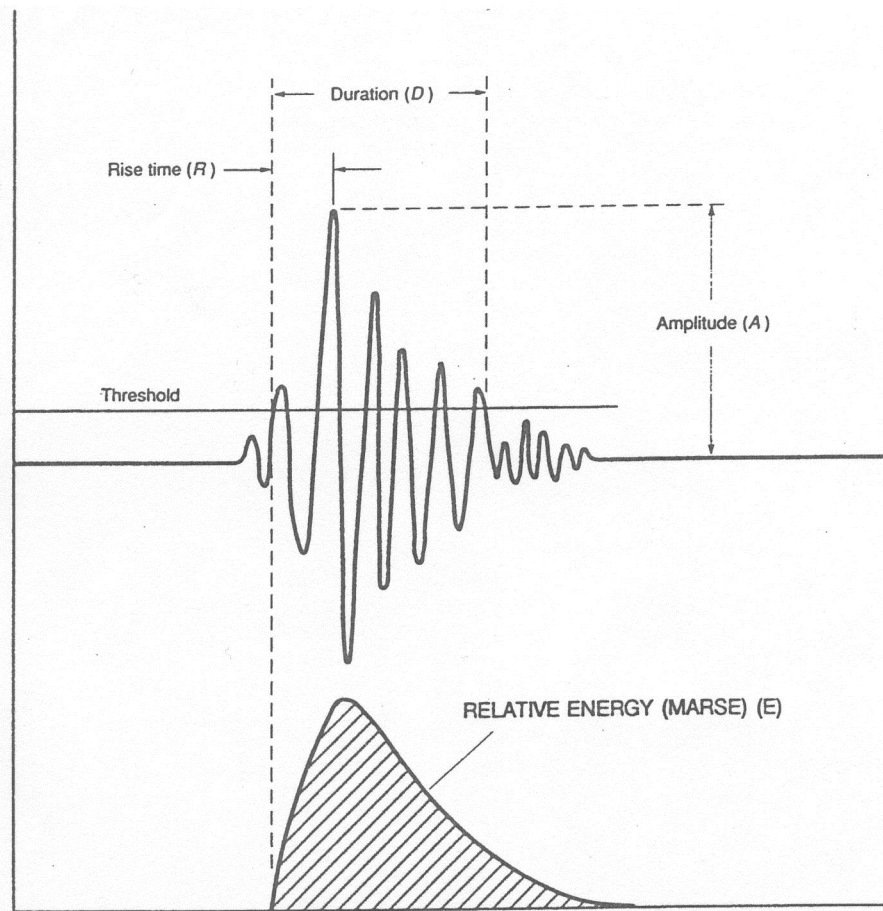


Fig. 2. Acoustic parameters of an event containing six counts. (Any type of pulse crossing the threshold is registered as a count [11]).

specimens were then reloaded with the second and third loads, which both stressed the specimen 15–20% higher than the preload. In the second and third cycles the load was removed rapidly after the peak [9]. The test carried out by Hunt et al. [9] and the current authors employed different specimen sizes and load saturation times, the latter being in our case four minutes. At the same time, the loading rate used was similar in the tests of both. All 21 mm samples were instrumented with two AE transducers and an axial extensometer kit. AE transducers were mounted with aluminum waveguides and silicon grease was used as a coupling agent. Figure 3 shows the complete instrumentation.

In addition to the LRR tests, two 32 mm specimens (#9 and #11) were loaded up to failure in order to investigate the uniaxial compressive strength in a direction perpendicular to foliation. These tests were based on the ISRM

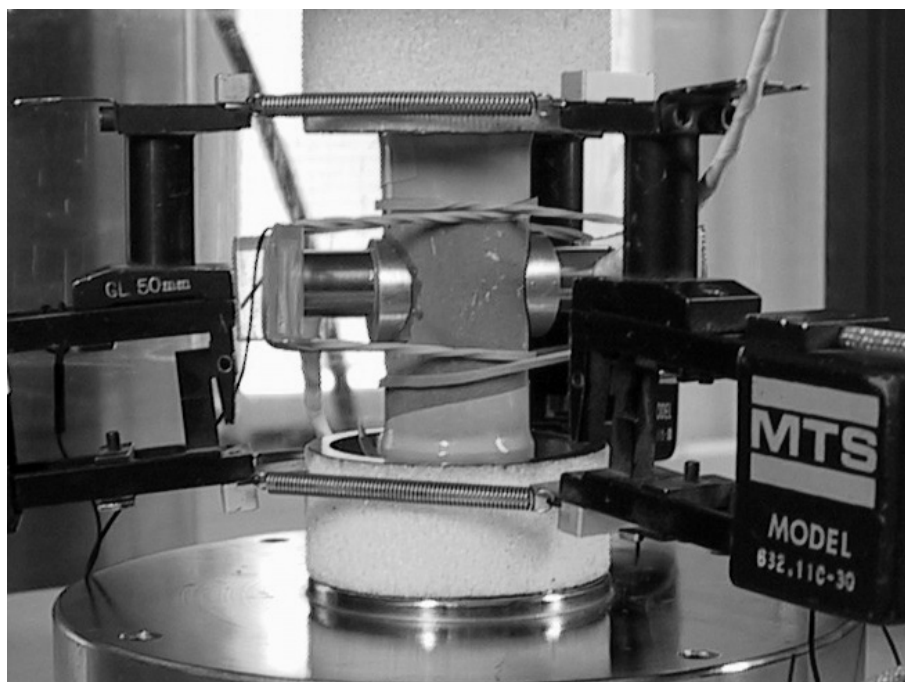


Fig. 3. An instrumented specimen prior to the test. The steel discs on the specimen ends are spacers for the 42 mm end pieces of the system.

suggested second-series method for determining the complete stress-strain curve for intact rock in uniaxial compression [10]. Specimen #9 was tested under axial strain control, initially with a strain rate of 200 microstrains per second. When the stress level reached 70% of the estimated ultimate strength, the strain rate was decreased to 20 microstrains per second. The second test was performed under circumferential strain control, with a similar change in lateral expansion rates. Acoustic emission was monitored during both these tests.

The first four cores (21 mm diameter) were tested within a week from preparation, and the other four were kept in storage for 6 months before testing. The moisture content of all cores was 1–3% during the experiment.

Being very distinct at a stress level of 21 MPa, the Kaiser effect was not observed during the second reloading.

## 5. Results

The results below can be useful to continue research on pillars long-term stability in conditions of Estonian oil shale mines for designing new underground mines. By laboratory tests pillars lifetime in case of changes in geological conditions can be determined.

Two out of the first four LRR tests were unsuccessful; the reloading of specimen #2 had a peak stress of 10.5 MPa, which was only 0.5 MPa higher than the preload stress. Thus, too few events were recorded to give reliable information on the Kaiser effect. The specimen was, however, totally quiet in the reloading phase, suggesting that the stress might have been memorized by it.

The second partially successful test was done on specimen #4. In this test the specimen fractured along the foliation planes during the first reload cycle at a 17 MPa stress level. The Kaiser effect broke down as well, the slope of the AE curve changed at 4 MPa during the reloading and no change of the slope was detected at the preload stress level of 15 MPa. The failure of specimen #4 was evident already in the preload cycle in which a macroscopic failure developed at a stress of 11.5 MPa. The failure was not visible on the surface of the specimen, but it was revealed by both the strain and AE data. The AE and strain graphs obtained in successful tests are shown in Figures 4–7.

The tests on specimens #3 and #6 gave better results. Specimen #3 displayed the KE at 21 MPa, which gives a Felicity Ratio of 95% with a preload stress of 20 MPa. It has to be noted that during the third loading cycle the specimen was totally quiet, suggesting that the first reloading was memorized by the sample.

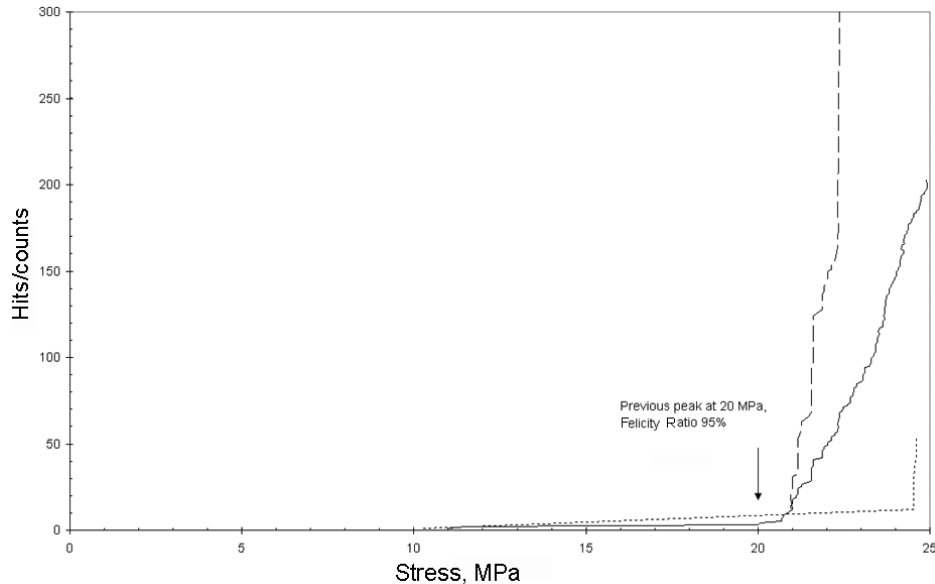


Fig. 4. Cumulative acoustic emission graph of specimen #3 during reloading (— hits, -- counts, - - - reload 2).



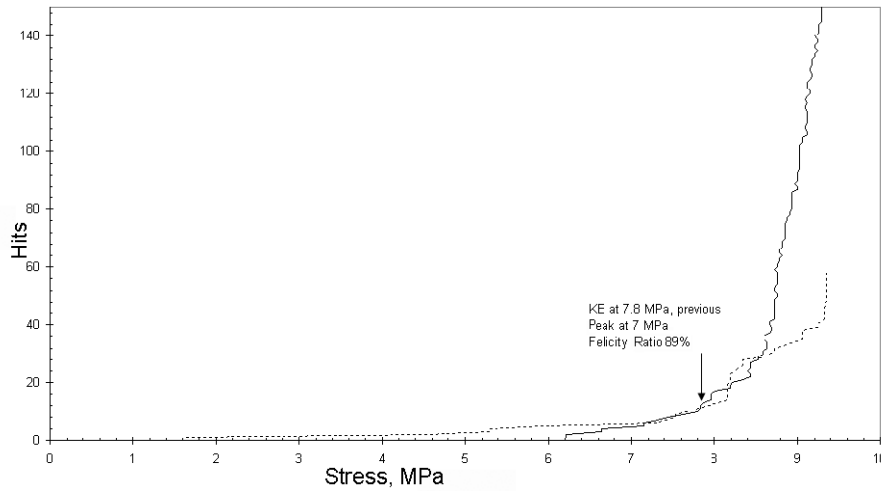


Fig. 5. Acoustic emission graph of specimen #6, with KE shown at 7.8 MPa. Noteworthy is the preservation of KE in the second reloading, as opposed to specimen #3 (— first cycle, - - - second cycle).

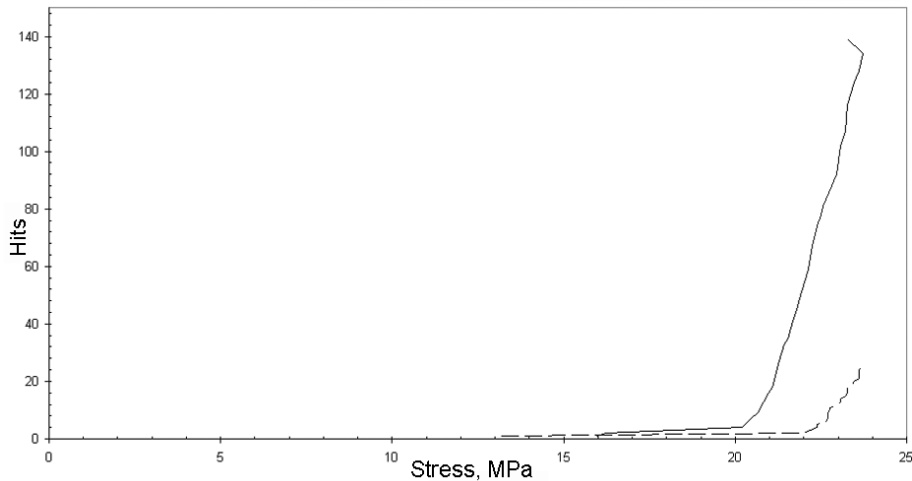


Fig. 6. A sorted acoustic emission graph of specimen #10, with a clear Kaiser effect at 20 MPa (— first reload, - - second reload).

Specimen #6 showed the Kaiser effect at a stress level of 7.8 MPa, which gives a FR of 89% with a preload stress of 7 MPa. Between 8 and 10 MPa, the emission rate accelerated to that of a stage associated with the macroscopic rupture of the specimen. The specimen seemed to be intact after the test, but closer inspection revealed that it was fractured in the direction of foliation.

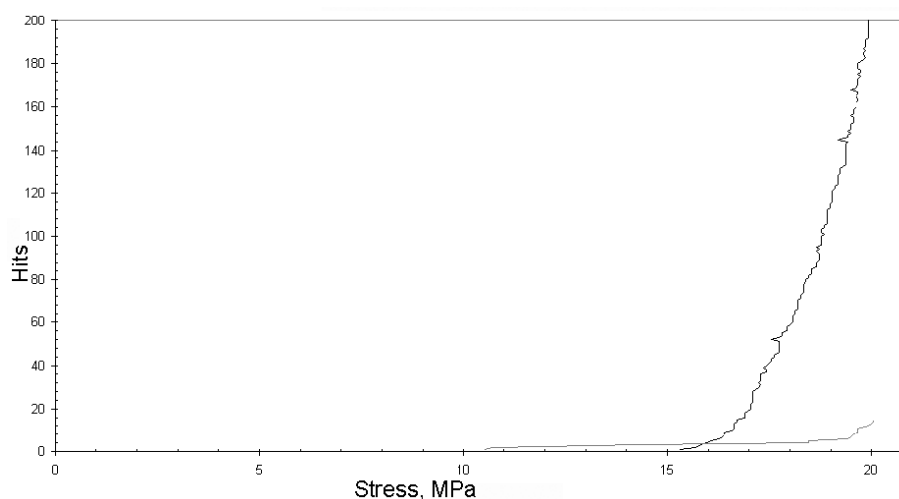


Fig. 7. Acoustic emission graph of specimen #12, with a clear Kaiser effect in the first reloading; the preload peak stress was 15 MPa (— first reload, — second reload).

The results of LRR tests done on 32 mm samples were similar to those obtained for 21 mm samples, so the sample size did not influence the Kaiser effect as expected. For specimens #10 and #12 the Kaiser effects were of better quality, the Felicity ratios being 100% and 94%, respectively. A delay time of one hour was used between the pre- and reloading of specimen #12, which had no significant effect on the stress memory.

The peak strengths recorded in two uniaxial compression tests done on specimens #9 and #11 were 35.5 and 40.5 MPa, respectively. Both the specimens failed abruptly, specimen #9 by multiple shear-dominated fractures intersecting at the mid-height of the specimen (hour-glassing).

The switch of control mode caused a small gap in data. In this test, the extensometers were attached to the sample, this arrangement produced much more linear strain data.

The peak preload stress level of specimen #10 was similarly 20 MPa. KE was not preserved in the second reloading, it moved to the 22 MPa stress level.

Specimen #12 was tested for KE with a one-hour delay between the pre- and reloadings.

Specimen #11 failed in an explosive manner with a multiple fracture locus near the top of the specimen [12]. Acoustic emission results showed that the crack initiation stress of oil shale was 15–17 MPa, in a direction perpendicular to foliation. No compressive strength tests were performed in a direction parallel to foliation.

## 6. Discussion

The performed LRR tests demonstrated that stress memory existed in oil shale, although the low strength of the material limited its usability. The tests also showed that when the preload stress approached the ultimate strength of the material, the Kaiser effect (memory) started to break down. This is characteristic of brittle rocks. The effect of delay time was not properly studied; after one hour, the Kaiser effect was still nearly perfect. According to Li [13], KE was still present in Kuru granite after a one-month delay. However, as oil shale and brittle rocks totally differ in mechanical nature, no suggestions can be made at this point.

The existence of the Kaiser effect in oil shale was confirmed, but measuring the in-situ stresses would require intact samples large enough for sub-sampling in multiple directions.

Another property of oil shale that might bring complications is its mechanical anisotropy. Specimen #4 showed a multiple tensile fracture during the first reloading at a stress level of 16.5 MPa, while the preload stress was 15 MPa [12]. The stress-strain curve of this specimen dropped at 11.5 MPa during the preloading. Specimen #6 entered the unstable fracture propagation stage during the first reloading at 8.5 MPa, but stayed intact during the test. These two tests confirmed the existence of the horizontal transverse isotropy of oil shale, suggesting that the strength of oil shale in a direction of foliation could be less than half its strength in a direction perpendicular to foliation.

The sorted acoustic emission graph of specimen #12 demonstrated the existence of a clear Kaiser effect in the first reloading; the previous peak stress was 15 MPa (Fig. 7). This specimen was tested for KE with a one-hour delay between the pre- and reloadings. The acoustic emission results showed that the crack initiation stress of oil shale was 15–17 MPa, in a direction perpendicular to foliation. The unconfined compressive strength of oil shale was 37 MPa [14].

Based on Equation (2), the time-dependent strength decay factor  $k_t$  reached its limit value of 0.44 when the pillars service life  $t$  approached infinity ( $t \rightarrow \infty$ ). If  $k_t < 0.44$ , the creep of rock will occur [2, 3]. As results from the acoustic emission tests, the crack initiation stress level was at  $k_t = 0.43$ . On the basis of the results obtained for specimen #9 (35.5 MPa) and specimen #11 (40.5 MPa) by using Equation (2), the long-time durability for both was 16.7 MPa [14]. This compared to the results obtained when using data given in Table 1, with  $R_t$  reaching 17.6 MPa.

## 7. Conclusions

The acoustic emission results showed that the crack initiation stress of oil shale was 15–17 MPa, in a direction perpendicular to foliation. The uncon-

finned compressive strength of oil shale was 37 MPa. Based on Equation (2), with  $k_t < 0.44$ , the creep of rock would occur. As resulted from the acoustic emission tests, the crack initiation stress level was at  $k_r = 0.43$ .

Based on the above results, the acoustic emission method can be used to estimate the long-term rock durability for pillars in conditions of Estonian oil shale mines. To verify the reliability of the method, however, further investigation of the Kaiser effect in Estonian oil shale and limestone will be required, in addition to trial measurements. The trials should preferably be performed on samples taken from the rock outside the mining zones, e.g. exploration drill cores.

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