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Mineral composition of shales and the results of triaxial compression tests – a case study from the Ordovician and Silurian rocks of Poland

Hydraulic fracturing is needed to extract hydrocarbons from shale rock formations. The efficiency of fracturing depends on many factors, but the mechanical properties of rocks are one of the most important of them. This paper presents the results of studies of the influence of the mineral composition of rocks on their mechanical properties. The investigated cores represent different formations from the Silurian-Ordovician basin from the polish part of the East–European Platform. X-ray diffractometry (XRD) and X-ray fluorescence (XRF) methods were used to establish the composition of the studied rocks, whereas the mechanical parameters were derived from triaxial compression tests. Correlation was conducted between the mineral composition and the mechanical parameters. The X-ray microtomography (micro-CT) method was used in order to visualize the structural changes in the rocks caused by triaxial compression. As a result of the research, a discussion on the influence of the rock matrix composition on its mechanical properties is presented.

Key words: shale gas, microtomography, brittleness, triaxial compression test.

Skład mineralny łupków a wyniki testu trójosiowego ściskania – na przykładzie skał sylurskich i ordowickich z NE Polski

Szczelinowanie hydrauliczne jest niezbędnym zabiegiem w celu wydobywania węglowodorów z formacji skał łupkowych. Efektywność szczelinowania zależy od wielu czynników, między innymi od właściwości mechanicznych skał. W artykule przedstawiono wyniki badań nad wpływem składu mineralnego skał na ich właściwości mechaniczne. Badane rdzenie reprezentują utwory syluru i ordowiku z polskiej części skłonu platformy wschodnioeuropejskiej. W celu określenia składu próbek wykorzystano metodę dyfraktometrii (XRD) i fluorescencji (XRF) rentgenowskiej, a parametry mechaniczne próbek skał uzyskano z wytrzymałościowych testów trójosiowego ściskania. W artykule opisano korelacje uzyskane pomiędzy składem mineralnym, a parametrami mechanicznymi. Dodatkowo wykorzystano metodę rentgenowskiej mikrotomografii komputerowej (micro-CT) w celu wizualizacji zmiany struktury próbek skał spowodowanej testem trójosiowego ściskania.

Słowa kluczowe: gaz z łupków, mikrotomografia, kruchość, testy trójosiowe.

Introduction

Recently in Poland there has been strong interest in the research on shale gas formations, which has been expressed in many newspapers and internet publications. Hydraulic fracturing process is needed to extract hydrocarbons trapped in such rocks. The efficiency of fracturing depends among others also on the mechanical properties of rocks [8]. The rocks' mechanical properties are derived from the mineralogical composition, degree of compaction and the internal structure in macro- and micro- scale and they can be measured directly [1, 5, 16].

There are several laboratory methods used for evaluation of mechanical properties of rocks. The main and straight forward method is the triaxial shear test [11]. Rock susceptibility to hydraulic fracturing can also be estimated based on the analysis of rock chemical or mineralogical composition, which are indicators of rock brittleness [6, 10].

This paper describes a complex study of the mechanical properties of rock performed on cores representing shales of Ordovician and Silurian age from various lithostratigraphic units from the polish part of the East European Platform. For comparison, samples from intercalations occurring within the shale complex (marls, limestones) have also been tested.

The aim of this research was to compare results of standard tests of the mechanical properties in rock cores with the mineralogical and chemical indicators of rock brittleness achieved by the X-ray diffraction and X-ray fluorescence methods. As a complementary method, a micro-computed tomography was applied in order to visualize the internal core structure before and after the triaxial compression tests. The observed changes were correlated with the lithological properties of the studied rocks.

Methodology

The investigated samples were cored from original rock cores. The samples were cylindrical in shape, approximately 8 cm in height and 5 cm in diameter (Figure 1).

The samples were subjected to different research methods in the following order:
- Micro-CT scanning,
- 3 axial compression test,
- Micro-CT scanning,
- X-ray fluorescence,
- X-ray diffraction.

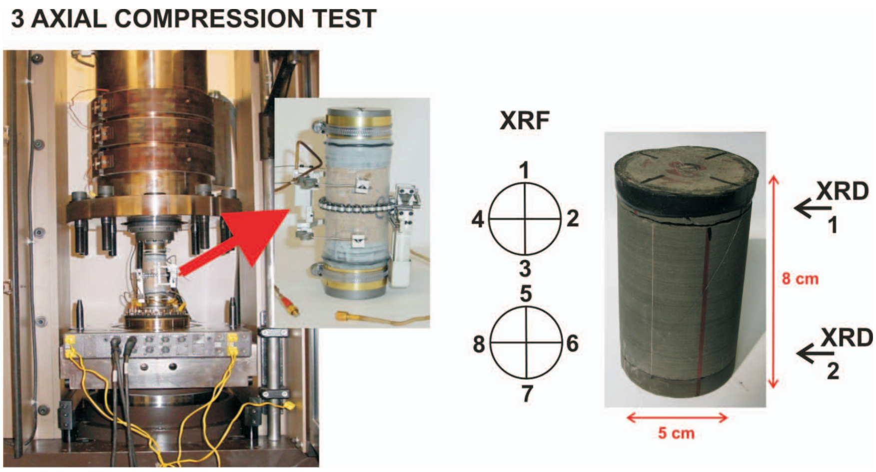


Fig. 1. Samples prepared for 3 axial compression tests and the scheme of the XRD and XRF measurements

Microtomography scanning

Micro-CT is a non-destructive and non-invasive method which allows to visualize the internal structure of the investigated object. More information describing the foundations of this method can be found elsewhere [13, 15]. Microtomography (micro-CT) tests were performed with the use of Nikon Benchtop CT160Xi apparatus in order to visualize the core structure changes caused by triaxial compression tests. Each sample was scanned twice, before and after the triaxial compression tests. In order to detect cracks after compression tests, the author's algorithm for crack detection was applied.

Triaxial compression tests

The aim of the conducted strength tests was first of all the determination of the elasticity parameters: Young's static elasticity modulus (E) and static Poisson's ratio (v). The compressive strength tests in the triaxial state of stress were conducted using the single failure test procedure. They were carried out according to the recommendations of the International Society for Rock Mechanics (ISRM) [4]. The triaxial compression tests and interpretation of the results were performed using MTS-815 device equipped with thermos-pressure chamber. The tests were conducted according to procedures of the MTS company, which were based on the ASTM 2664-95a [17] standard, as well as the recommendations of the ISRM's *Suggested Method for Determining the Strength of Rock Materials in Triaxial Compression*.

In this paper the main focus was put on the values of the Young's static modulus (E) as well as Poisson's static ratio (v).

The Poisson's ratio was defined as the relation between the size of the axial (ε_z) and lateral (ε_{xy}) strains; for further analysis we selected Poisson's ratio for the full linearity of all the characteristics of deformation (v_{avl}) corresponding to the ASTM and ISRM recommendations, however it was determined in more detail, only to the limit of microdilatancy (microcracking) established according to the phenomenological description by Hallbauer and others [2]. For Young's modulus we selected the average modulus (E_{av}), which is closely connected to the phenomenological conduct of rock sample deformation and appears to be the most representative for the measured rocks.

X-Ray Diffraction (XRD)

Quantitative X-Ray Diffraction (XRD) analysis was conducted by the Rietveld method with the use of the SIROQUANT program [7, 9, 14], which is useful for analyzing samples containing clay minerals. The quantitative measurements were carried out on Panalytical X'Pert Pro apparatus with modern ultra fast detector (real time ultra strip X'Celerator). Voltage 40 kV and current 40 mA were applied, samples were scanned from 5° to 65° 2Θ. Preparations were made according to the method introduced by Środoń et al. [12]. Zincite (ZnO) was added to the samples as an internal standard. Samples were grinded for 5 minutes in the McCrone micronizing mill in order to reduce the grain size to <20 µm. Internal standard (ZnO) was added prior to grinding to obtain full homogenization of the samples. Measurements were conducted on random preparations made by side-loading.

X-Ray Fluorescence (XRF)

X-Ray Fluorescence (XRF) chemical analysis was conducted with the use of the BUKER S1 TITAN, a fully field portable analyzer based on energy dispersive XRF (EDXRF) technology. The S1 TITAN uses an Rh target X-ray tube (max: 50 kV, 100 µA, 4 W) and 10 mm² X-Flash® Silicon Drift Detector (SDD) (typical resolution of 147 eV at Mn K-alfa peak). Applied X-ray spectrometer works with Fundamental Parameters (FP) software to allow for elemental quantification of completely unknown samples without standards. The device enables elemental analysis for the range from magnesium (Mg) through uranium (U).

Rock brittleness, defined as the susceptibility of rocks to fracturing, is usually determined by its mineralogy – as quartz content [6] or by chemical composition – as SiO_2 content [10]. In the case of the Silurian-Ordovician rocks from Poland the mineralogical indicator of brittleness was defined as a sum of quartz and feldspar.

Results

Nine core samples representing different lithostratigraphic formations have been investigated. The samples for the tests were selected based on sedimentological analysis of cores and log analysis. The selected samples represent lithostratigraphic units of the Ordovician (Prabuty marls and claystones formation, Sasino claystones formation, Kopalino limestones formation) and the lowermost Silurian (Pelplin claystones formation, Pasłęk claystones formation, Jantar bituminous claystones member). These units correspond to the stratigraphic interval between the Arenigian and the Wenlockian. Rocks from the clay formations are similar in terms of mineralogical composition, structure and texture. They differ in colour (light grey to black) and organic carbon content (TOC). Besides shales, samples of marl and limestone were also included to the investigation.

The XRD confirmed the differences in the mineral composition of the studied rocks. Table 1 presents selected mineral

Table 1. Results of mechanical and mineralogical measurements

Sample no.	Brittleness as a SiO_2 content (XRF)	Brittleness as a sum of quartz and feldspars content (XRD)	Sum of carbonates (XRD)	Sum of clays (XRD)	Poisson's static ratio (v)	Young's static modulus (E_{av})
	[wt%]	[wt%]	[wt%]	[wt%]	[-]	[GPa]
1	64.27	26.7	13.1	55.0	0.19	30.0
2	66.83	27.9	1.3	66.5	0.17	21.7
3	44.73	17.3	14.5	47.6	0.12	23.6
4	63.46	32.5	22.0	44.0	0.19	38.6
5	22.55	7.6	71.8	18.3	0.26	50.4
6	68.17	28.8	2.5	66.3	0.16	22.8
7	67.71	28.2	3.1	66.8	0.15	18.3
8	70.65	29.2	8.8	59.7	0.16	26.9
9	64.97	27.7	2.8	57.9	0.12	15.1

composition results as well as Poisson's ratio and Young's modulus values.

A correlation analysis was performed for the parameters listed in Table 1. Examples of results are shown in Figure 2. Young's modulus values and Poisson's ratio are highly dependent on the rock mineralogical composition. Young's modulus values are higher for higher carbonate concentrations (Figure 2a). For Poisson's ratio a good correlation was observed with the clay minerals content. Poisson's ratio values are lower for the higher clay minerals content (Figure 2b). The correctness of the mineralogical analysis was proved by the XRF analysis (Figure 2c and 2d).

For comparison on the Figure 3 the obtained data was placed on the diagrams illustrating the lithological diversity in the whole basin scale. On the first plot two different correlation curves describe the relation between the SiO_2 content and the mineralogical composition – in that case with the sum of quartz and feldspars. The change in the slope of the trend line is the result of the presence of SiO_2 also in the clay minerals structure. The SiO_2 content in clay minerals structure is about 40÷50%, much less than in quartz and feldspars, but it starts to be important for the clay reach samples. In the range of dominance of quartz and feldspars in mineralogical composition of samples the slope of trend line is controlled by chemical composition of those two minerals. With the rise of clays content it changes accordingly to the chemical composition of clay minerals present in the studied material.

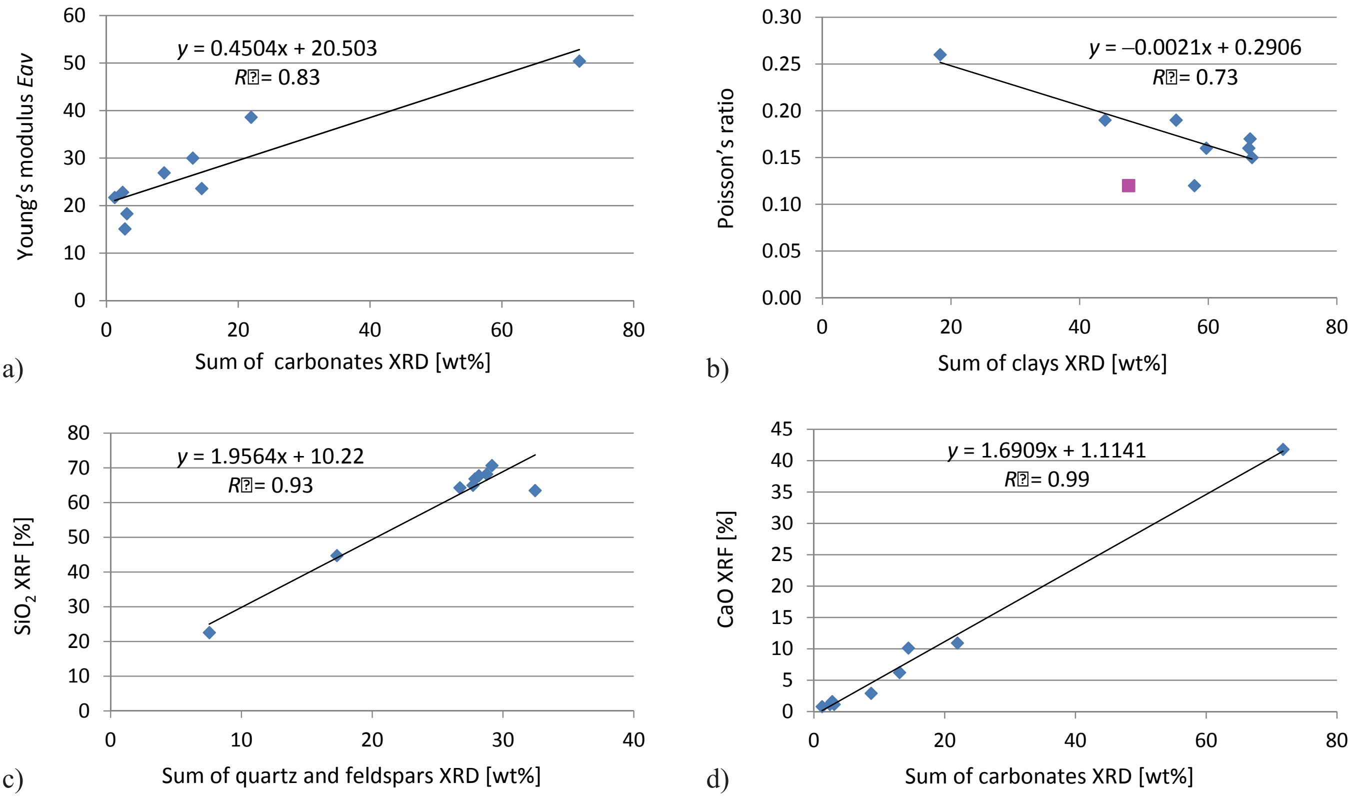


Fig. 2. Correlations of the obtained parameters. Pink dot on the plot b) – represent the results obtained for the bentonite sample

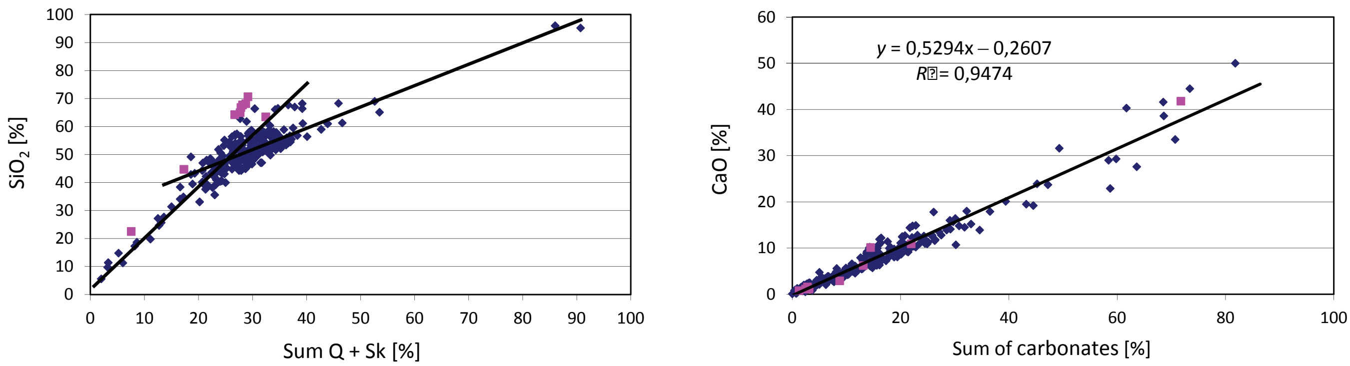


Fig. 3. Mineralogy of the analysed samples (pink colour) comparing to the data set representing whole Silurian–Ordovician basin lithological diversity

Micro-CT results

According to the micro-CT results, the investigated cores may be divided into two groups. The first group includes samples with minor structure changes and the second group represents samples containing big cracks extending throughout the whole core structure. To the first group belong two samples with the highest concentration of carbonates (samples 4 and 5).

The second group samples show some tectonics cracks in their structure. These tectonic cracks could be the main reason why the visualization of the second group samples showed the most expanded crack after the triaxial compression test. Figure 4 presents examples of the first and second group samples. The top example shows the group one sample and the bottom example refers to samples representing group number two.

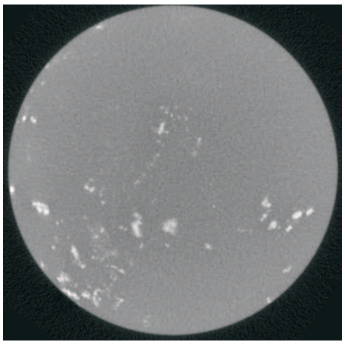
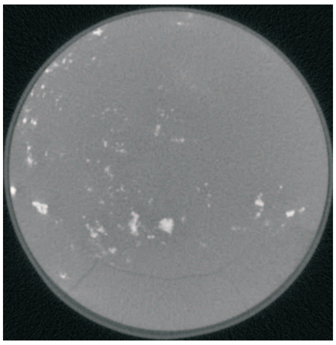
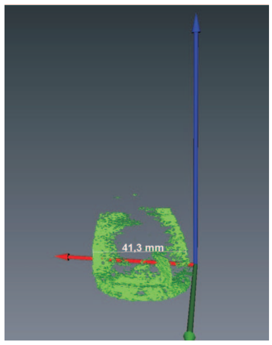
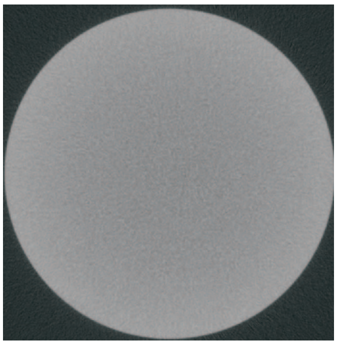
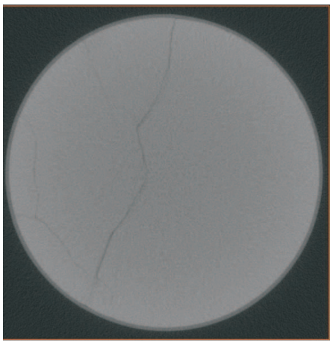
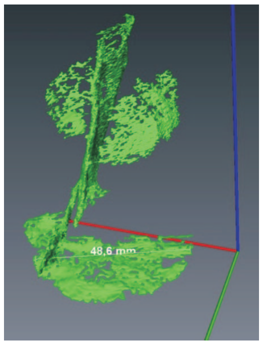
Sample group no.	Cross-section before the triaxial compression test	Cross-section after the triaxial compression test	3D visualization of core structure changes
1			
2			

Fig. 4. Visualization of the core structure changes after the triaxial compression test

Conclusions

The correlation of triaxial compression and mineralogical tests (XRD and XRF) results have shown the presence of a strict relation between the mechanical rock properties and their mineralogical composition. It has been concluded, however, that the carbonate content (strict correlation with Young's modulus obtained in mechanical tests) and the sum of clay minerals (strict correlation with the Poisson's ratio) are better indicators of brittleness of the Ordovician and Silurian rocks from the Polish basin than standard one. The SiO_2 content or the quartz content (sum of quartz and feldspars), used as the brittleness indicator in other basins, has a restricted application in the Polish basin. Because the Ordovician and Silurian rocks reveal very good correlation with the content of carbonates determined by the XRD method and the CaO content using the XRF method, it seems that XRF measurements may aid in fast assessment of the brittleness of the studied medium. On the other hand the tomographic data revealed that for the investigated clay-silt core samples, the cracks that appeared during the compression tests followed tectonic structures of the investigated rocks. The micro-CT results showed the necessity of sampling very homogeneous samples, because the presence of tectonic cracks could influence the results of deformational tests, as it is the second, main factor determining the mechanical properties of rock [3].

Please cite as: Nafta-Gaz 2015, no. 6, pp. 355–360

Article contributed to the Editor 17.02.2015. Approved for publication 17.04.2015.

Acknowledgments

The authors of this paper are grateful to PGNiG SA for the possibility to use this data. Micro-CT, XRF and XRD tests were performed at the Oil and Gas Institute – National Research Institute. Triaxial compression tests were performed at University of Warsaw at the Institute of Hydrogeology and Engineering Geology. This paper was presented at the Annual Symposium of the Society of Core Analysts at 2014 in Avignon, France.

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