# Engineering of experiment

**Chevychelov V.Yu., Korneeva A.A.**

**Evaluation of the oxygen fugacity ($f_{O2}$) in experiments in internally heated pressure vessel (IHPV).** UDC 550.42

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**Abstract.** Experimental method of buffer associations is used to evaluate the gas regime of complex reactions occurring at elevated *P* and *T*. Two-capsule technique, first proposed by G. Eugster, is usually used for monitoring $f_{O2}$. The oxygen fugacity ($f_{O2}$) in experiments in internally heated pressure vessel ("gas bomb") was evaluated at $T$= 1000°C and $P$= 200 MPa using various solid oxygen buffers (Co-CoO, Ni-NiO, $Fe_3O_4$-$Fe_2O_3$, Cu-$Cu_2O$). These buffers were chosen according with paper I.M. Chou [1987], and the $f_{O2}$ values for these *P-T* parameters were calculated using the formula $\log(f_{O2})_{P,T} = A/T+B+C(P-1)/T$. The calculated $\log(f_{O2})_{P,T}$ were equal (in atm.): -11.86 for Co-CoO; -10.23 for Ni-NiO; -5.48 for $Fe_3O_4$-$Fe_2O_3$ and -6.02 for Cu-$Cu_2O$. The experiments were carried out in sealed double Pt capsules. A buffer mixture was placed in the inner capsule, and $H_2O$ was loaded in the external capsule to increase the work time of the buffer. The initial buffer mixtures were composed in such proportions as: 85 wt.% Co (Ni) and 15 wt.% CoO (NiO); 15 wt.% $Fe_3O_4$ (Cu) and 85 wt.% $Fe_2O_3$ ($Cu_2O$). The duration of the experiments was 1 day. After the experiments X-ray diffraction analyses of buffer mixtures were conducted. It was found that in all the experiments, both components of the buffer were retained, but their ratios changed significantly. In the experiments with Co-CoO and Ni-NiO, the quantity of oxidized phases increased for account of more reduced ones. In experiments with $Fe_3O_4$-$Fe_2O_3$ and Cu-$Cu_2O$, in contrast, the quantity of more reduced phases increased. Thus the value of log ($f_{O2}$) is in the range about -10 ÷ -6 or Ni-NiO + ≈2-3 in experiments in our "gas bomb" at $T$= 1000°C and $P$= 200 MPa. Our results coincide with the data, log $f_{O2}$ ≈Ni-NiO+3.5, reported in paper J. Berndt et al. [2005].

***Keywords:*** *oxygen buffer; oxygen fugacity; experiment; internally heated pressure vessel (IHPV); buffer association.*

Experimental method of buffer associations [Nekrasov et al., 1982] is used to evaluate the gas regime of complex reactions occurring at elevated *P* and *T*. Two-capsule technique, first proposed by G. Eugster [Eugster, 1957], is usually used for monitoring $f_{O2}$.

The oxygen fugacity ($f_{O2}$) in experiments in internally heated pressure vessel ("gas bomb") was evaluated at $T$= 1000°C and $P$= 200 MPa using various solid oxygen buffers (Co-CoO, Ni-NiO, $Fe_3O_4$-$Fe_2O_3$, Cu-$Cu_2O$). These buffers were chosen according with paper [Chou, 1987], and the $f_{O2}$ values for these *P-T* parameters were calculated using the formula $\log(f_{O2})_{P,T} = A/T+B+C(P-1)/T$. The calculated $\log(f_{O2})_{P,T}$ were equal (in atm.): -11.86 for Co-CoO; -10.23 for Ni-NiO; -5.48 for $Fe_3O_4$-$Fe_2O_3$ and -6.02 for Cu-$Cu_2O$.

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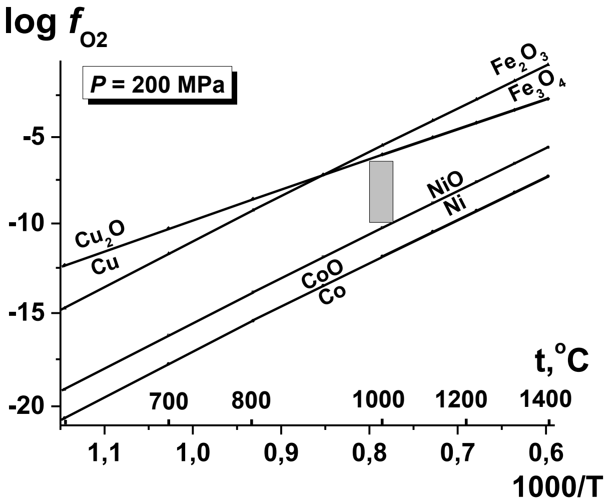


**Fig. 1.** Evaluation of the oxygen fugacity ($f_{O2}$) in experiments in internally heated pressure vessel (IHPV).

**Acknowledgments.** This study was financially supported by the RFBR, project № 18-05-01001A.

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## Lebedev E.B., Zevakin E.A., Zevakin D.E. Equipment on the basis of super-high-speed centrifuge for modelling and experimental researches of formation of metallic nucleus of Moon.

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**Abstract.** In detail described the equipment, that building on the basis of super-high-speed centrifuge and that allowing to carry out scientific and experimental researches, that linking separation of liquids and crystals in zones of partial melting, accumulation and precipitation of metal phase in conditions of melting of initial planetary substance. By means of such equipment one can to create modern technologies in order to receive of super-clean metals and alloys, and also of materials with special properties.

***Keywords:*** *equipment, super-high-speed, centrifuge, partial melting, gravitation, rotation, formation of Moon, compression, system, collector, generation and transmission of ultrasonic oscillations, rectification of metals alloys, accumulation.*

Super-high-speed centrifuge one may to use for modelling and research of process metallic nucleus of Moon and planetary bodes of earthly type formation. According to the very wide-spread hypothesis Moon was formed as a result of rapid rotation and strong gravitation of primary powdered cloud (Vinogradov,1975; Galimov,2004). At this time inside of compressing cloud the temperature begin to rice, happen such processes, as particle melting of primary matter, segregation of metallic phase in kind of melting drops and their accumulation in middle of primary cloud (Lebedev and other, 2016).

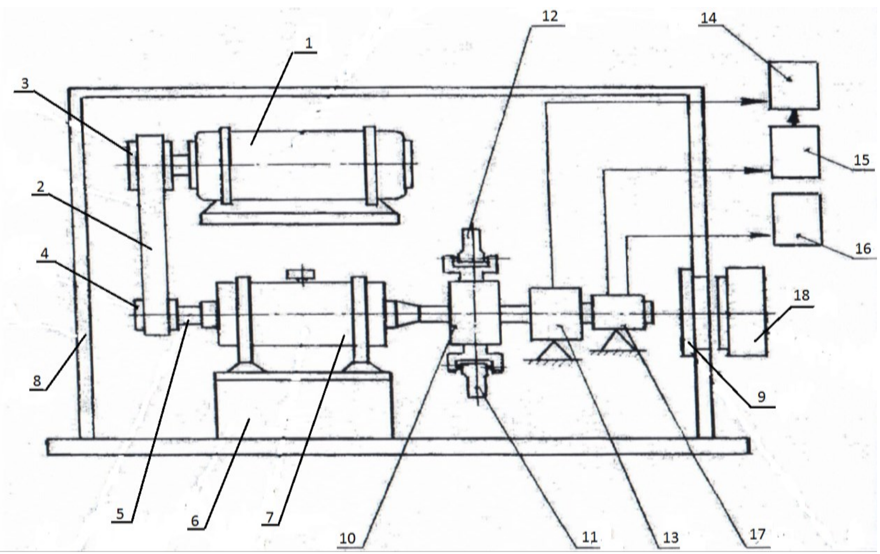
So, in order that to carry out modelling of process forming of planets, their nucleuses, it is necessary to create strong gravitation field and to ensure necessary temperature for particle melting of primary substance. These conditions one can to ensure by the use of super-high-speed, super-high-temperature centrifuge, as field of centrifugal forces creating by centrifuge, is analogue of gravitation forces, and temperature in object of research one can to create with help of electric heater.

Such equipment on the basis of centrifuge was projected, done and using at present by experimental researches at GEORYI RAS (Lebedev and other. 1997). The equipment is a system that allow to modelling and research of great number of natural phenomenons and process, which connected with forming of nucleus of Moon, magmatic activities of the Earth, to expose of conformity to natural lows of division of liquides and crystals in zones of partial melting (Kadik and other, 1989; Lebedev and other. 1996).

For increase the efficiency of modelling of such processes as separation of metallic melting from crystals, accumulation and falling of metallic phase in conditions of melting of initial planetary matter, it is necessary to ensure synchronous influence on melting as of centrifugal force and high temperature, thus and of percussion pulse load (of base-frequency ultrasonic oscillations).

Given task one can to solve by means of modernization of super-high-speed, super-high-temperature centrifuge by means of introduction in its system of block of ultrasonic oscillations. (Lebedev and other. 1989; Lebedev fnd other 2014)/

On figure 1 is represented scheme of describing eqnipment.




1-electric motor; 2-belt drive; 3,4-pulleys; 5-shaft; 6-base; 7-hinge support; 8-protective cabin; 9-inspection window; 10-cross-piece; 11-super-high-temperature furnace; 12-counterbalance; 13-copper-graphite collector; 14-block of feeding and regulation of furnace temperature; 15 – block of registration of furnace temperature; 16-block of feeding and control of ultrasonic oscillator; 17-mercury-platinum collector; 18 –stroboscope.

**Fig.1.** Scheme of equipment on the basis of super-high-speed and super-high-temperature centrifuge for modelling and experimental researches of the formation of metallic nucleus of the Moon.

The equipment works in follows way. The rotation from the electric motor 1 through the belt drive 2, that fixed on the driving 3 and driven 4 pulleys passing to the shaft 5, that fixed on a hinge support 7 with a fastened on it counterbalance 12. The centrifuge is located in the protective cabin 8, and the hinge bearingt 7 is fixed on the base 6. In protective cabin 8 is being one a protective window 9, through which the help of stroboscope 18 one can to determine by sight numbe of revolution of centrifuge.Feeding and regulation of temperature of revolving furnace realizing by means of block 14 through a copper-graphite collector 13. The construction of the copper-graphite collector (to revoling copper discs pressing springer copper-graphite brushes) is widely distributed and therefore is not provided here. Copper-graphite collector go up for transmissions of high currents (in this case, through such a collector passing current for heating of revolving furnace to a temperature of 1430-1450$^{o}$C). For feeding of the ultrasonic oscillator in the furnace and for register the temperature of the furnace (with the help of thermocouple), such a collector is not suitable, because it distorting of size of passing currents , while current's taking from thermo-couple, as and currents, that are necessary to control the ultrasonic oscillator, are very small and their distort cannot be tolerated, as other wise the results of research will be incorrect. Therefore, in this present case, the mercury-platinum collector 17 is the most suitable, as it practically do not distort of passing currents . It connects stationary disposed blocks 15 (block of registration of temperature in revolting furnace) and the 16 (block of feeding and control of ultrasonic oscillator that fitted in revolving furnaces 11) accordingly with thermocouple and the ultrasonic oscillator revolving with super-high-temperature furnace.

**Perspectives of application** By means of equipment on the basis of super-high-speed, super-high-temperature centrifuge with possibility of creation of ultrasonic oscillations and passing their influence in working zone one can considerably to raise efficiency of solution of series fundamental scientific problems, and also to create, work through and bring to perfection special break-through ultra-modern technologies, which allow to divide of super-high-temperature mixtures, that consist of crystalline fission phases, to receive of pure materials with high degree of homogeneity structure (for example, alloys, glasses with different compositions) and also materials with special characteristics for application in equipment and articles of special purpose.

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## Shikhova N.M., Tselmovich V.A., Patonin A.V. Interrelation of structure and acoustomechanical properties of rocks. UDC 550.34.016

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**Abstract.** Analysis of polished sections of sandstones with the use of digital micrographs was carried out. The indices of connectivity and self-similarity of structure of various types of sandstones, including kerogen-containing rock, are estimated. Examples of the distributions of acoustic activity arising during laboratory tests of these materials under conditions of one- axis deformation are considered. It is shown that the character of the distributions of acoustic activity is related to the structural features of the samples, the parameters of which are characterized by the results of microphotographic analyzes. This makes it possible to evaluate the change in the structure of the test sample at various stages of its stress-strain state.

***Keywords***: *rock, acoustic emission, image analysis, self-similarity, porosity, grain circularity.*

The formation of rock is due to the processes of precipitation, compression, destruction, cementation. In this case, particles and pores of different sizes are formed. The self-similarity of the rock structure can be characterized by a power-law dependence of the number of particles of a given size on their size. And this dependence is valid in a range of several orders of magnitude. At the same time, power dependences are known, which are manifested in the acoustic properties of rocks. In laboratory tests of rock samples in a controlled press INOVA under uniaxial and triaxial deformation recorded continuous stream of acoustic emission (AE) emitted by the sample. This stream contains information about the internal structure of the sample, the homogeneity of its grain, the nature of the porosity, fluid fullness. Since the conditions of rock formation (temperature, pressure, depth effect) are different, then different values of self-similarity index characterize these properties distributions. At the same time, a number of rocks do not possess the expressed properties of a self-similar (fractal) structure. The purpose of this paper is to analyze the structural characteristics of sandstone samples of various types and to reveal their relationship with the power parameters of the AE signals distributions.

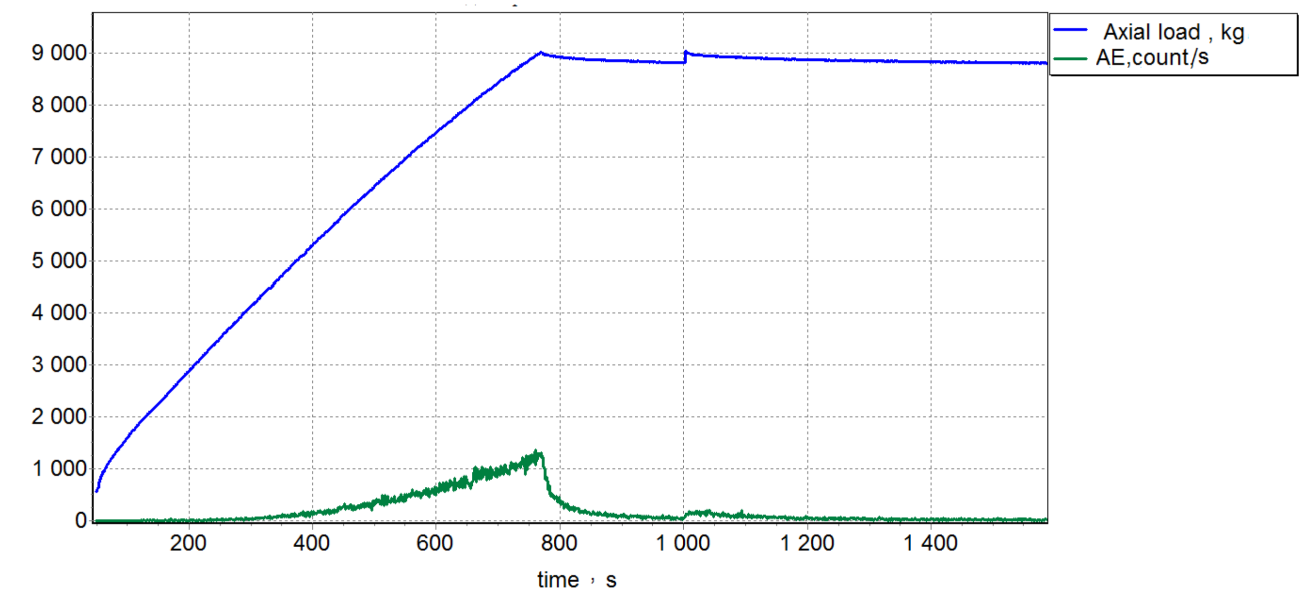



**Fig.1**. Typical load characteristic in testing on INOVA press.

**Material and methods of analysis.** Three sandstone samples of different types differing in the porosity and characteristic grain sizes (Table 1) were used. The test samples were shaped into cylinders with the dimensions of 60 mm in length and 30 mm in diameter . One of the sandstones contained kerogen. Experiments on the selected types of sandstones were carried out under conditions of axial loading with a constant strain rate of $10^{-6}$ 1/s (Patonin, 2006). A fragment of the load curve of one of the experiments is shown in Fig 1. As a result of processing the AE stream, an acoustic event bulletin is formed that contains the amplitude, energy and time of each identified event. The total number of acoustic events recorded during the experiment can reach $15X10^6$ with a resolution of up to 5000 events per second. Statistical processing of the bulletin included the calculation of the self-similarity of the flow of acoustic events in a sliding time window of 120-300 s.

To analyze the bulletin of AE signals, the initial load section was selected before the formation of the main fault. At this stage, there is a collapse of the pores initially present in the sample, partial consolidation of the material, without dilatancy and mutual slippage of individual grains. This stage can be characterized as elastic with a small plasticity. As the strain increases, the intensity of the acoustic emission flow increases to 1000 events per second. Polished section of of the starting materials were analyzed using a scanning electron microscope TESCAN VEGA II.

The processed photomicrographs were processed in the ImageJ package (https://imagej.net). The ratio of the area of the pores to the total area of the image was calculated, the value obtained was taken as the porosity of the sample (Kadyrov, Zakirov, 2016). The fractal Hausdorff dimension (D) of pore space was calculated by the cell cover method (Feder, 1991). The degree of roundness of the grains was estimated by the factor Circ = $4*\pi*S/p^2$, where S- is the area and p-is the perimeter of the grain. The dependence of the number of grains (N) on their linear dimensions (L) was approximated by a power function of the form $N \sim L^{-c}$, the dependence of the number of AE events on their energy by the power function $N \sim E^{-b}$. The coefficients c, b for approximations with a confidence level of at least 0.9 were estimated using the method of least squares.

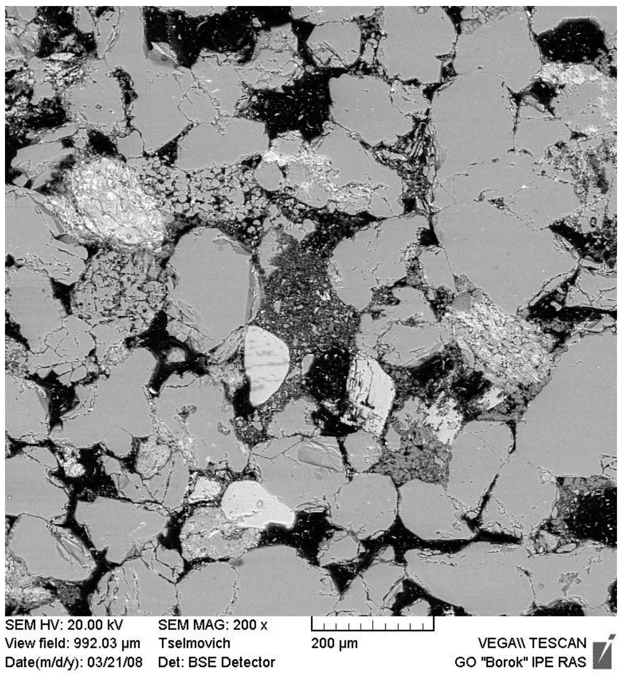



**Fig.2.** A photomicrograph of sandstone Berea polished section

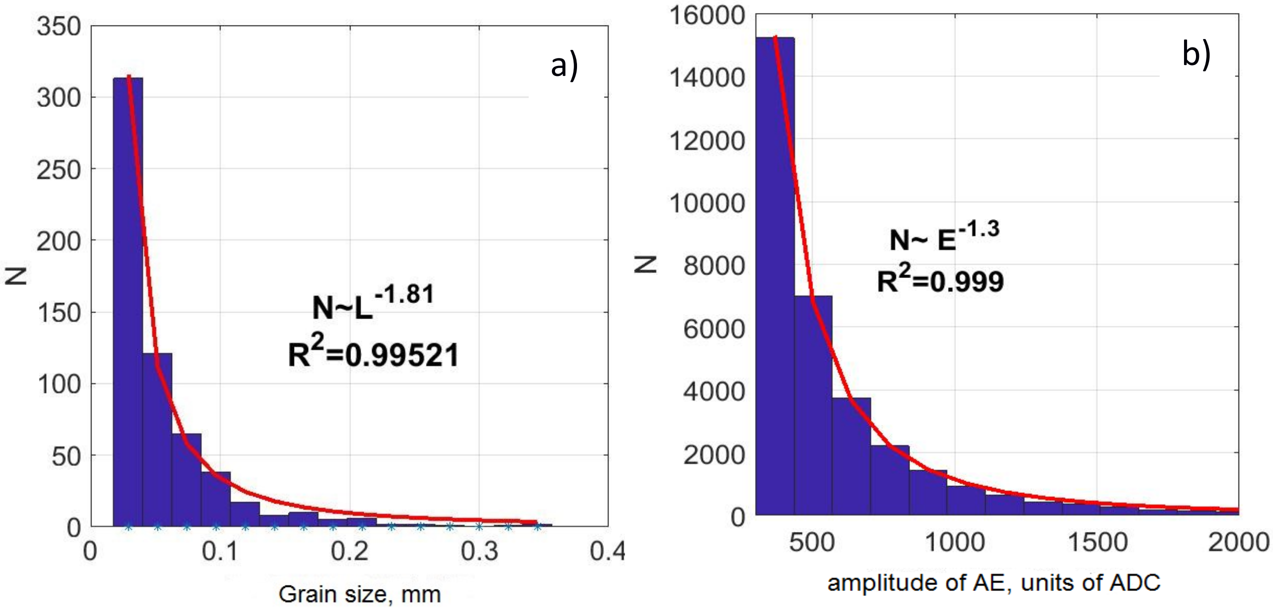



**Fig.3.** a) the grain size distribution, b) the amplitude istribution of the acoustic events of Berea sandstone.

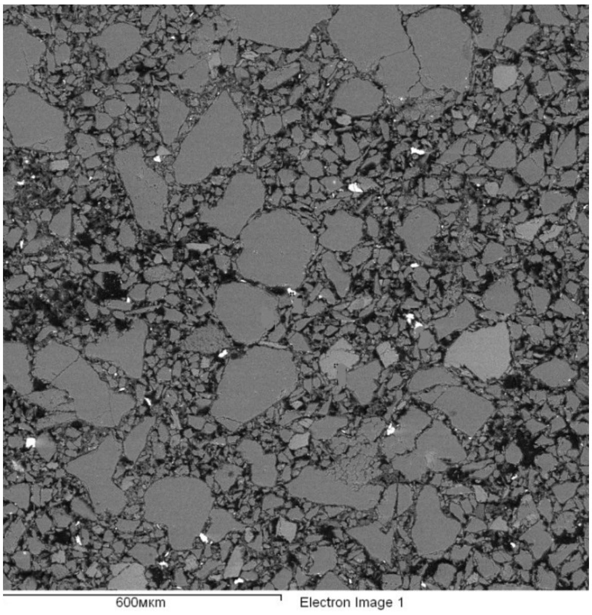
**Table 1.** Characteristics of the structure and acoustic properties of sandstones

| Sandstone type | Calculated porosity | Roughness of grains | c±Δc | b± Δb | D± ΔD |
|---|---|---|---|---|---|
| Berea | 23.37% | 0.63±0.006 | 1.81±0.07 | 1.3±0.03 | 1.67±0.12 |
| Kerogen | 8.31 % | 0.3±0.004 | 2.23±0.6 | 1.17±0.06 | 1.29±0.16 |
| Castlegate | 19.53% | 0.49±0.008 | 1.94±0.25 | 1.09±0.07 | 1.61±0.12 |

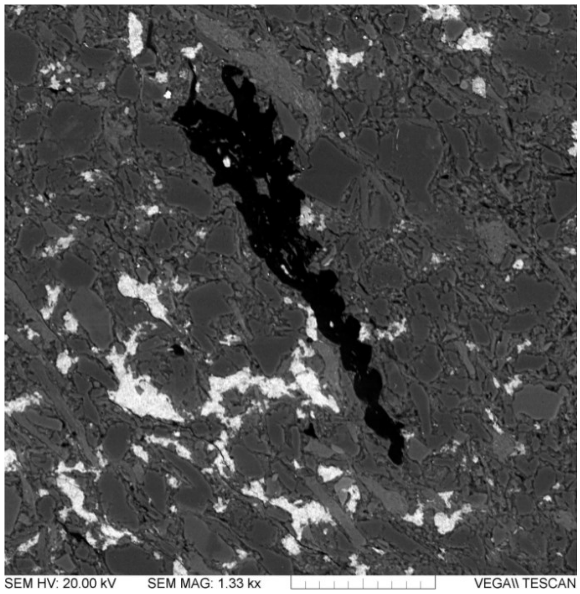
**Results and discussion.** The analysis results of polished sections and acoustic emission flow are summarized in Table 1. Figure 2 shows a photo of the polished section of Berea sandstone. Gray color shows the grains that make up the structure of the sandstone matrix, black colour porous space. The distributions of Berea grains in size are shown in Fig. 3a. The validity of the approximation of this distribution by the power function N ~ $L^{-c}$ can be characterized by the determination coefficient, the value of which is close to 1, and the standard error Δc, with c/Δc << 3. Note that for sandstones of two other types, the power distribution of grains by size is statistically less validate. Analysis of the amplitude distribution of acoustic events (Fig. 3b) showed that the largest value of the value of the exponent b in the initial stages of the experiment was found for the Berea sandstone (Table 1), that is, the contribution of acoustic events with a smaller amplitude to general AE in this type of sandstone is significantly greater than in the two others. In this case, the accuracy of the approximation of acoustic distributions by a power law dependence for all sandstones is equally high. The structure of the three types of sandstone is different: the grain size distribution (c) in Berea is the smallest, which indicates a higher uniformity in the distribution of grain sizes of different linear dimensions, with the Berea grains being the largest of the three types of sandstone. It can be assumed that when Berea is loaded in the process of destruction of a single grain, a few small ones form, while in a similar process in Castlegate sandstone, when one grain is destroyed, only two smaller grains, close in size, usually form.

Photomicrographs of Castlgate and Kerogen sandstones are shown in Fig.4a and Fig.4b, respectively. Calculation of the porosity of the material, made using microphotographs, showed that the values obtained for the total porosity coefficients for these types of sandstones differ from those obtained by instrumental measurements by no more than 1.15 times. The values of the fractal dimension of the pore space of the Berea and Castlegate sandstones are close, which is an indicator of the same magnitude of the positive connectivity of their pore space (Filatov, 2006). The connectivity of the same Kerogen sandstone, calculated on fractal dimension of pore space, is negative, due to the presence of large amount of isolated pores.

**Conclusion.** On the basis of digital images, an analysis of the polished sections of rock samples has been carried out to calculate the porosity parameters. The indicators of connectivity and self-similarity of materials of different structures, including kerogen-containing rocks, are estimated. The indexes of the distributions of acoustic activity arising in the course of laboratory tests of rock samples of different structure in the axial deformation are analyzed. It was revealed that the character of the distribution of acoustic activity is related to the peculiarities of the structure of sandstones, discovered as a result of the analysis of polished sections. This allows us to predict the dynamics of destruction of the test sample at various stages of its stress-strain state. It is shown that the characteristics of the Berea sandstone (the highest values of porosity, the statistically significant power distribution of grains in size, the large roundness of the grains, the positive connection of the pore space) also determine the values of the power distribution of acoustic events at the initial loading stage higher than for other sandstones. It is assumed that peculiarities of the structure of pore space of sandstone containing kerogen (low porosity, negative connections of porous space - pore isolation, weak roundness of grains, etc.), cause a wide range of changes in parameters b and c and their weak interrelation.




a)




b)

**Fig.4**. A photomicrograph: a) Castlegate, b) Kerogen polished section

**Acknowledgments.** The work was carried out within the framework of state. № 0144-2014-0096 "Physics of transient and trigger processes in seismicity: laboratory modeling, field observations, petrophysical analysis", No. AAAA-A17-117040610183-6 "Spatio-temporal structure of the ancient geomagnetic field and petrophysics of magnetic minerals as indicators of geological and geophysical processes " and with the support of the RNF grant 16-47-02-003.

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