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ИССЛЕДОВАНИЕ ПОРИСТОСТИ ОБРАЗЦОВ ОСАДОЧНЫХ ПОРОД МЕТОДАМИ ЯДЕРНОГО МАГНИТНОГО РЕЗОНАНСА И ЛАЗЕРНО-УЛЬТРАЗВУКОВОЙ СТРУКТУРОСКОПИИ

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Аннотация: Исследовались образцы осадочных пород одного из месторождений Тимано-Печорской нефтегазоносной провинции. Проведен петрографический анализ образцов на сканирующем электронном микроскопе, работающем в режиме оптического изображения, определены геометрические параметры пор, видимых на поверхности образца. Установлено, что минеральный состав представлен вторичным доломитом и вторичным доломитом известковистым. На поверхности образцов наблюдались поры с размерами от 1,5 до 35 мкм и кавернами от 43 до 1750 мкм. Методом гидростатического взвешивания определена открытая пористость образцов, значения которой лежали в диапазоне от 9,5 до 13,40%. Полная пористость определялась методами ядерного магнитного резонанса (ЯМР) и лазерной ультразвуковой структуроскопии. Для ЯМР использовался релаксометр GeoSpec+ 2/75, измерялось время поперечной релаксации, по которому определялась полная пористость. В лазерной ультразвуковой структуроскопии рассчитывалось среднее значение скорости продольной волны в беспоровой среде при заданном минеральном составе, а затем по измеренным скоростям также рассчитывалась полная пористость. Получено, что найденное значение полной пористости обоими методами в среднем на 1% выше, чем открытой. Разница в значениях полной пористости, измеренных двумя методами, составляла не более 0,2–0,4%.

Ключевые слова: пористость, доломит, петрофизические свойства, ядерный магнитный резонанс, время поперечной релаксации, лазерная ультразвуковая структуроскопия, скорость волны, сканирующая электронная микроскопия.

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Study of porosity of sedimentary rock samples by nuclear magnetic resonance and laser-ultrasound diagnostics

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Abstract: The paper describes a study of sedimentary rock samples from an oil and gas field of the Timan-Pechora Basin Province. Petrographic analysis of the samples was conducted by using a scanning electron microscope operating in an optical image mode, the geometry of surface pores characterized. The mineral composition was represented by secondary dolomite and secondary calcareous dolomite. Pores 1.5 to 35 μm in size and caverns 43 to 1750 μm long were observed on the surface of the samples. The open porosity of the samples was estimated by using the hydrostatic weighing method, ranging from 9.5 to 13.40%. The total porosity was determined by using nuclear magnetic resonance (NMR) and laser-ultrasound diagnostics. A GeoSpec+ 2/75 analyzer was used to measure the transverse relaxation time so as to estimate the total porosity. The longitudinal wave velocity in a non-porous material with the same mineral composition was calculated and then the total porosity was estimated. It is found that the total porosity determined by both methods is on average 1% higher than the open porosity. The difference in the total porosity measured by the two methods is no more than 0.2–0.4%.

Key words: porosity, dolomite, petrophysical properties, nuclear magnetic resonance, transverse relaxation time, laser-ultrasound diagnostics, wave velocity, scanning electron microscopy.

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Introducton

Understanding rock-fluid interaction in porous media is very important to the mining industry [1—3]. Properties of rocks and saturating fluids are usually determined to decide which reservoir development method to employ [4, 5]. Such important petrophysical properties as porosity, permeability, capillary pressure, etc. are estimated not only from borehole logging measurements, but also must be supplemented from laboratory experimental data [6]. One of the methods to determine the aforesaid properties is nuclear magnetic resonance (NMR) that is used in borehole logging and for calibration of the measurement data in laboratory [7, 8].

The basic principle of NMR is that all nuclei with non-zero spin have magnetic moments that are aligned when affected by an external magnetic field, creating macroscopic magnetization in fluid-saturated rock specimens. NMR signals are generated by fluids (oil, water, or brine) when the rock sample placed in a magnetic field is excited by a short radio-frequency electromagnetic pulse. Immediately after the pulse, an NMR signal arises and then decays with time due to relaxation, which is characterized by the relaxation time of the NMR signal (decay time) known as the transverse relaxation time T_2 [9].

The amplitude of the NMR signal observed immediately after the pulse is an indication of the total amount of fluid present, while T_2 provides information about the fluid and the size of pores it fills. Short relaxation times (100 μs or less) indicate that the samples have small pores while long decay times come from fluids in large pores [10]. A mathematical operation known as inversion is used to extract several components in the relaxation time, which carry information on different fluids [11]. Further processing of the electromagnetic pulse observed, including the use of gradient fields and other methods (e.g. water saturation method, centrifugation, etc.), ma-

kes it possible to more accurately determine the chemical composition of the fluids and evaluate wettability, permeability, capillary pressure, and pore size distribution [12, 13].

This paper addresses examination of sedimentary rock samples from an oil-and-gas field of the Timan-Pechora Basin Province, including petrographic analysis by optical methods and scanning electron microscopy to characterize the geometry of surface pores. The open porosity of the rock samples was determined by the water saturation method and the total porosity, by NMR. The results were verified by using laser-ultrasound diagnostics.

Methods and materials

A group of cylindrical samples of carbonate rocks were examined, three of which are shown in Figures 1, 2 and 3. The samples were taken from producing formation D3el of a field of the Timan-Pechora Basin Province. The samples were about 30 mm in diameter and length. At first oil was extracted and then the samples were dried to remove residual fluids.

The mineral composition and the structure and size of surface pores were evaluated by using a ThermoScientific Quattr. S scanning electron microscope operating in an optical imaging mode.

NMR experiments were performed by using a GeoSpec$^+$ 2/75 analyzer generating static and pulsed magnetic fields. Porosity was determined using a well-known method [14] that is based on the fact that macroscopic magnetization of liquids in pores relaxes after the magnetic field is removed. The smaller the pore, the shorter the transverse relaxation time T_2. Micropores 1–10 μm in size are characterized by the relaxation time less than 1 ms while mesopores (10–100 μm) and macropores

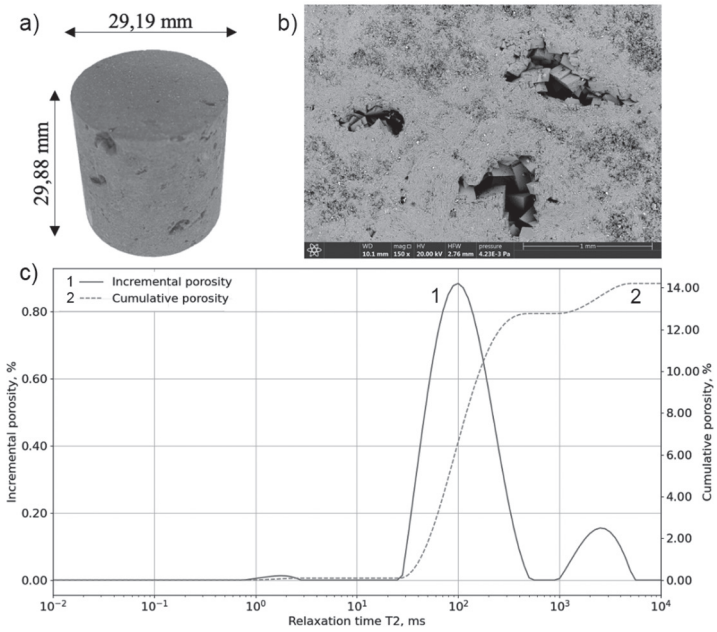

Fig. 1. Sample No. 1: dolomite sample (a); SEM image of dolomite sample topography (b); NMR transverse relaxation time curves vs. NMR transverse relaxation time curves (c)

Рис. 1. Образец № 1: фото образца доломита (а); оптическое изображение, полученное на СЭМ, поверхности образца доломита (б); зависимость пористости образца доломита от времени поперечной релаксации по ЯМР (в)

(0.1–1 mm) are characterized by T_2 ranging from 1 to 10 ms and from 10 to 100 ms, respectively. It is shown in [14–16] that the transverse relaxation time T_2 is proportional to the characteristic pore size r (1).

$$r \sim \rho T_2, \quad (1)$$

where ρ is the relaxation activity of the liquid phase (μm/ms), which has been measured for 250 fluids [14].

Therefore, after relaxation curve approximation, relaxation times are estimated, which determine the characteristic sizes of pores and their percentage by the signal amplitude corresponding to the respective relaxation time after the applied fields are removed.

The preliminarily dried samples were saturated with water and placed in the analyzer. The transverse relaxation time T_2 and signal amplitudes were measured. Pore size distribution and total porosity vs. relaxation time diagrams were plotted through the use of the analyzer software.

Local elastic wave velocities were measured by laser-ultrasound diagnostics in a pulse-echo mode and porosity was determined from the measurements [17–19].

It is known that if porosity is less than 20–25% the following relationship is valid [20]:

$$\frac{V_l}{V_{l0}} = \left(1 - P^{\frac{2}{3}}\right)^{\frac{1}{2}} \quad (2)$$

where V_l is the longitudinal ultrasonic wave velocity in the sample, ms; V_{l0} is the longitudinal ultrasonic wave velocity in an ideal material with no pores, m/s; P is the porosity of the sample.

Thus, if porosity is expressed in terms of longitudinal wave velocity V_l (3) and averaged over several points for each sample, the total porosity can be determined.

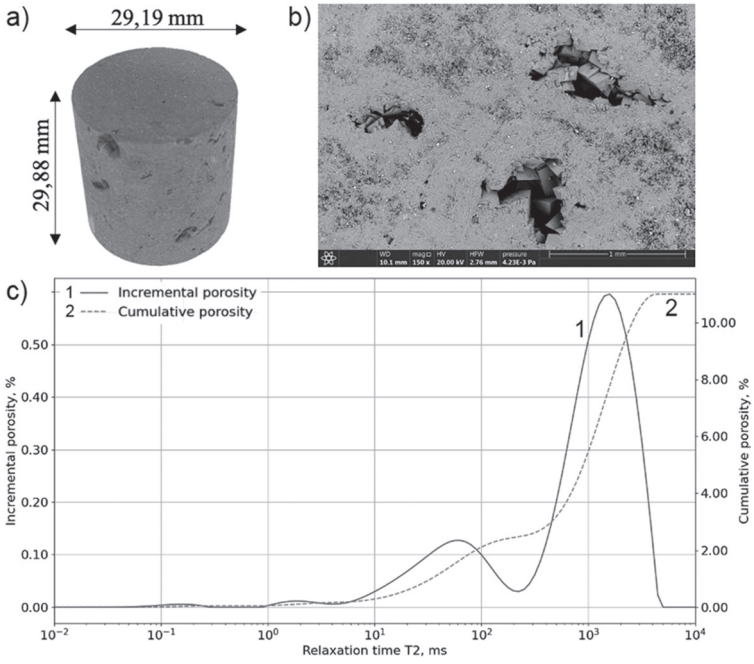

Fig. 2. Sample No. 2: dolomite sample (a); SEM image of dolomite sample topography (b); NMR transverse relaxation time curves vs. NMR transverse relaxation time curves (c)

Рис. 2. Образец № 2: фото образца доломита (а); оптическое изображение, полученное на СЭМ, поверхности образца доломита (б); зависимость пористости образца доломита от времени поперечной релаксации по ЯМР (в)

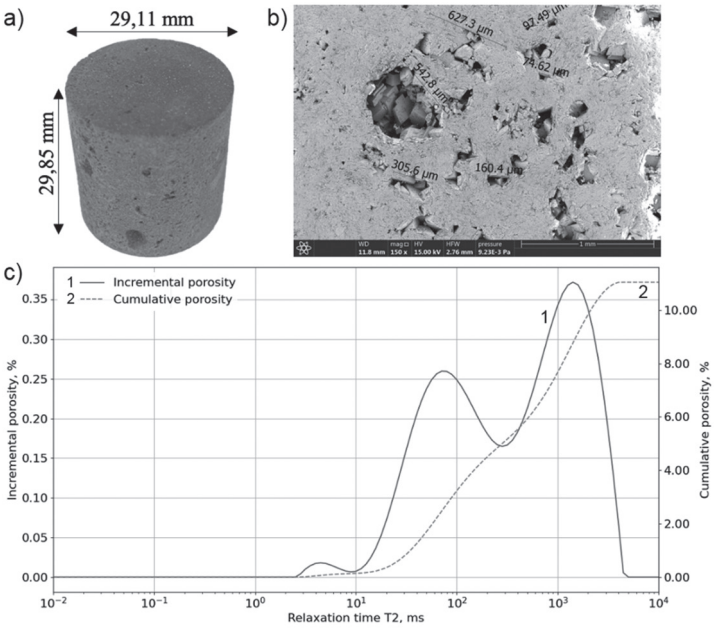

Fig. 3. Sample No. 3: dolomite sample (a); SEM image of dolomite sample topography (b); NMR transverse relaxation time curves vs. NMR transverse relaxation time curves (c)

Рис. 3. Образец № 3: фото образца доломита (а); оптическое изображение, полученное на СЭМ, поверхности образца доломита (б); зависимость пористости образца доломита от времени поперечной релаксации по ЯМР (в)

$$P = \left(1 - \left(\frac{V_l}{V_{l0}}\right)^2\right)^{\frac{3}{2}} \qquad (3)$$

Results and discussion

Optical studying has shown that the samples are composed of secondary dolomite and secondary calcareous dolomite. The samples are characterized by high porosity with pores 1.5 to 35 µm in size and cavities 43 to 1750 µm long (Figs 1, 2, and 3).

Hydrostatic weighing of the samples has shown that the open porosity ranges from 9.5 to 13.40% (Tab. 1). Determining total porosity by NMR implies investigating how the incremental porosity (solid curves in Fig. 1 to 3) and the total porosity (dashed curves in Fig. 1, 2, and 3) are related to the transverse relaxation time T_2. Since the characteristic pore size is directly proportional to T_2 (1), it is the pores with larger characteristic sizes r that contribute more to the observed NMR signal as the relaxation time increases (Fig. 1, 2, and 3). The total porosity ranges from 11.00% to 14.41% (Tab. 1) and on average is 1% greater than the open porosity determined from hydrostatic weighing. Note that the second and third samples have approximately the same total porosity (11.00% and 11.04%, respectively) and differ in incremental porosity distribution. Thus, the maximum incremental porosity of sample 1 corresponds to T_2 ~0.1 s, which means that the characteristic size of pores is about 0.1–1.0 mm while cavities are several mm in size (determined by T_2 ~1.0–1.5 s), making an insignificant contribution to the porosity.

The maximum incremental porosity of sample 2 is observed at transverse relaxation time $T_2 > 1$ (Fig. 2), which suggests that there are many cavities several mm in size.

Porosity of samples of secondary medium-to-fine-grained dolomite determined by different methods
Результаты определения пористости образцов доломита вторичного средне-мелкозернистого различными методами

Sample No.	Porosity determined by hydrostatic weighing, %	Porosity from NMR, %	Porosity measured by laser-ultrasound diagnostics, %	Longitudinal wave velocity, m/s
1	13.40	14.20	14.41	5657
2	9.50	11.00	11.38	5811
3	10.06	11.04	11.19	5821

Laser-ultrasound diagnostics was used to verify the total porosity determined from NMR measurements. The samples were cut into disks about 5 mm thick; the disks were scanned by using an ultrasonic scanning instrument. Longitudinal wave velocity was measured at several points.

In accordance with [19 — 21], given that dolomite has a trigonal system, known coefficients of the stiffness matrix [22] were used to determine quasi-longitudinal wave velocity in dolomite with no pores as the average of the velocities in six directions. The resulting velocity is 6643 m/s; the average longitudinal wave velocities measured by laser-ultrasound diagnostics are given in Table. The total porosity (table) is determined from the resulting velocity values. The porosity based on laser-ultrasound diagnostics data differs from NMR porosity by 0.21% for the first sample, by 0.38% and 0.15% for the second and third ones, respectively. This indicates that the total porosity determined by NMR is well in agreement with that determined by laser-ultrasound diagnostics.

Conclusions

The paper presents experimental data on porosity of reservoir rock specimens taken from an oil field of the Timan-Pechora Basin Province. Three methods were employed to perform experiments: (1) hydrostatic weighting, (2) NMR, and (3) laser-ultrasound diagnostics. It is found that the total porosity is on average 1% greater than the open porosity. The difference in the total porosity measured by NMR and laser-ultrasound diagnostics is no more than 0.4%. Therefore, the laser-ultrasound diagnostics method may well be more effective than NMR due to the high cost of equipment and labor costs for NMR research as far as the total porosity is concerned.

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