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The Potential Applications of Electron Microscopy in Studying the Lithological Characteristics of Oil-Bearing Sandstones

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Возможности применения метода электронной микроскопии при изучении литологических особенностей нефтеносных песчаников

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The purpose of the research is to apply a technique for studying of the void space in potentially oil-bearing reservoirs using electron microscopy. Microscopic studies of characteristic areas of the core surface are carried out with an assessment of the void space microstructure, the characteristics of the fragmentary framework grains and filler, and the microstructure of the cementing substance. The surface of the samples is scanned at different magnifications, and microprobe analysis is carried out to diagnose the grains of the detrital framework, filler, cementing and newly formed substances.
The relationship between the filtration and capacitance properties of reservoirs and the characteristics of the granulometric composition, void space, composition and structure of cement has been studied.
A number of groups have been identified in the Solikamsk Depression reservoirs, for which a regular relationship between productivity and the features of granulometric composition, void space, composition and structure of cement has been established. The first group of reservoirs is characterized by good sorting and dense packing of quartz detrital framework grains, a small amount of filler particles and cementing substance represented by kaolinite with "book-like structure" type. Reservoirs with reduced permeability are characterized by an increased proportion of cementing substance, poor grain sorting due to the presence of a significant amount of filler particles in the detrital framework. A characteristic feature of such a reservoir is a less perfect microstructure of kaolinite of the "book-like structure" type. For the studied siltstones, an mixed-grained granulometric composition, no grain sorting by size, unstructured kaolinite cement with no "book-like structure" type have been established.
The lithological differences of sandstones of the same geological age, determining their oil-bearing capacity, are determined by the important role of the facies environment of sedimentation and subsequent processes of rock transformation at the stages of diagenesis and catagenesis. The electron microscopy method can be recommended for wider application in oil and gas geology when assessing the lithological features of oil and gas reservoirs.

Ключевые слова:
месторождения нефти и газа, электронная микроскопия, нефтеносные песчаники, пористость, проницаемость, литология.

Целью исследований является применение методики изучения пустотного пространства потенциально нефтеносных продуктивных пластов методом электронной микроскопии. Проведены микроскопические исследования характерных участков поверхности керна с оценкой микроструктуры пустотного пространства, особенностей зерен обломочного каркаса и заполнителя, микроструктуры цементирующего вещества. Выполнено сканирование поверхности образцов при разных увеличениях, микрозондовый анализ с целью диагностики зерен обломочного каркаса, заполнителя, цементирующего и новообразованного вещества.
Изучена связь фильтрационно-емкостных свойств коллекторов с особенностями гранулометрического состава, пустотного пространства, состава и структуры цемента.
В составе коллекторов Соликамской впадины выделены группы, для которых установлена закономерная связь продуктивности с особенностями гранулометрического состава, пустотного пространства, состава и структуры цемента. Первая группа коллекторов характеризуется хорошей сортировкой и плотной упаковкой зерен кварцевого обломочного каркаса, небольшим количеством частиц заполнителя и цементирующего вещества, представленного каолинитом со структурой типа «книжный домик». Коллекторы с пониженной проницаемостью характеризуются повышенной долей цементирующего вещества, слабой сортировкой зерен за счет присутствия значительного количества частиц заполнителя в обломочном каркасе. Характерным признаком такого коллектора является менее совершенная микроструктура каолинита типа «книжный домик». Для исследованных алевролитов установлен разнозернистый гранулометрический состав, отсутствие сортировки зерен по крупности, неструктурированный каолинитовый цемент с отсутствием структур типа «книжный домик».
Литологические различия песчаников одного геологического возраста, определяющие их нефтеносность, обусловлены важной ролью фациальной обстановки осадконакопления и последующих процессов преобразования породы на стадиях диагенеза и катагенеза. Метод электронной микроскопии можно рекомендовать для более широкого применения в нефтегазовой геологии при оценке литологических особенностей нефтегазоносных коллекторов.

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Introduction

The research objective is to apply a specific methodology for studying the void space of potential oil-bearing productive formations based on scanning electron microscopy data. The objects of the study are core samples of terrigenous oil deposits. Studying the rock structure by electron microscopy has certain limitations due to the small size of the studied samples, in which only the surface structure details (grains, voids, aggregates, etc.) are studied. At the same time, the method has certain advantages: additional data of the substance obtained at the micro-level of study, clear peculiar micro-objects visualization, the ability to determine the chemical composition of indicator inclusions by the microprobe method, etc.

A large number of works are devoted to the electron microscopy of terrigenous oil reservoirs. There have been considerable successes in studying the void space and cementing material under an electron microscope [1–3], as well as in examining secondary mineral formation processes [4–5].

In this study, the structures and void spaces of terrigenous reservoirs in the oil fields of the Solikamsk depression (Perm Krai) were analyzed using electron microscopy. The main objective was to establish lithological parameters and features identified by electron microscopy methods related to the productivity of rocks.

Problem statement. Materials and methods

The research object is the reservoirs of the Visean deposits in the Solikamsk depression, where several relatively large oil fields have been discovered in recent decades [6–8]. The Visean productive formation is the major one in Perm Krai, its oil content is connected with promising terrigenous reservoirs of the granular (porous) type. It should be noted that the industrial oil content formation of the Visean reservoir, located directly under the Verkhnekamsk potassium-magnesium salt field, has significantly contributed to the amount of oil reserves in the region. Currently, the prospects of the Visean reservoir are estimated to be very high. In this regard, a number of studies have been carried out in Perm Krai to ensure rational complex development of salt and oil deposits [9–11].

For 33 Visean objects in the Solikamsk depression, the values accepted by the State balance for K_{por} range from 11 % to 17 % (on average – 14 %), and for k, from 5 to 655 mD (on average – 173 mD). The boundary values for granular type reservoirs, below which the rock is considered non-reservoir in reserve calculations, are accepted for objects with K_{por} in the range of 7 % to 10.6 %, and for k – from 0.75 to 2.5 mD. Overall, the wide range of variation in the porosity and permeability properties suggests a complex lithological structure of the productive deposits, characterized by alternating layers of different structures.

The core samples collection analyzed in the work is represented by 11 samples from four oil fields (Sibirskoye, Unvinskoye, Shershnevskoye and Zhilinskoye) in a wide range of reservoir properties with variations in porosity coefficients (K_{por}) from 3.9 to 21.5% and permeability (k) from 0.3 to 749 mD. Samples with reservoir properties above the established boundary values for reservoirs are classified as sandstones.

According to the practice of Visean deposits development in the Solikamsk depression, among the reservoir properties, rock permeability has a decisive effect on oil displacement coefficients and well productivity [12–14]. The works [15–17] demonstrate that fluid entry can occur from intervals that according to the data of a standard well logging complex, specifically the assessment of granular porosity, are classified as non-reservoirs. In this regard, an additional core sample of siltstone with reservoir properties below the boundary values (K_{por} < 4 %; k < 1 mD), was included in the research. It should be noted that for the study area, the oil fields are characterized by low viscosity (ranging from 0.7 to 3.5 mPa·s), which allows for productive well operation with relatively low reservoir properties.

Analytical studies were carried out at the Center for Collective Use with unique scientific equipment of Perm State National Research University – scanning electron microscope JSM 6390LV by Jeol (Japan). Microprobe analysis of objects was conducted with energy-dispersive spectrometer INCA ENERGY 350 (Oxford Instruments). The device operating mode was as follows: working voltage 30 kV, working distance 12 mm, and current strength 10 mA.

Characteristic areas of the core sample surface were photographed at different magnifications of the electron microscope. The main objects of the study were the microstructure of the void space, the features of the clastic framework grains and filler, the microstructure of the cementing material and new mineral formations. The microprobe method was used to determine the chemical composition of the components, and diagnostics were performed to identify the mineral types. The core samples were examined to study the sizes, shape of grains and packing of clastic particles, the nature and elements of the void space, the composition and microstructure of the cementing and secondary materials, which is the result of superimposed mineralization. Surface scanning of the samples was conducted at different magnifications (from tens to several thousand times), microphotography of characteristic structures and microobjects, microprobe analysis to diagnose the grains of clastic framework, filler, cementing material, and a new substance.

Study of void space in rocks by scanning electron microscopy methods

The void space of reservoirs is the main research object using electron microscopic methods [18, 19].

Microcavities. The largest forms of void space in the studied reservoirs are microcavities. Surface scanning of the sample was carried out at a microscope magnification of 50X to detect microcavities. The scanning showed that they are particularly widespread in core samples with high values of effective porosity (K_{por} – from 14.6 to 21.5 %; k – from 147 to 749 mD).

The occurrence of microcavities decreases significantly in core samples taken from sandstone interbeds with reduced porosity (11.4–13.6 %) and permeability (23.0–87.70 mD). Notably, microcavities are completely absent in the core of the densest sample (siltstone).

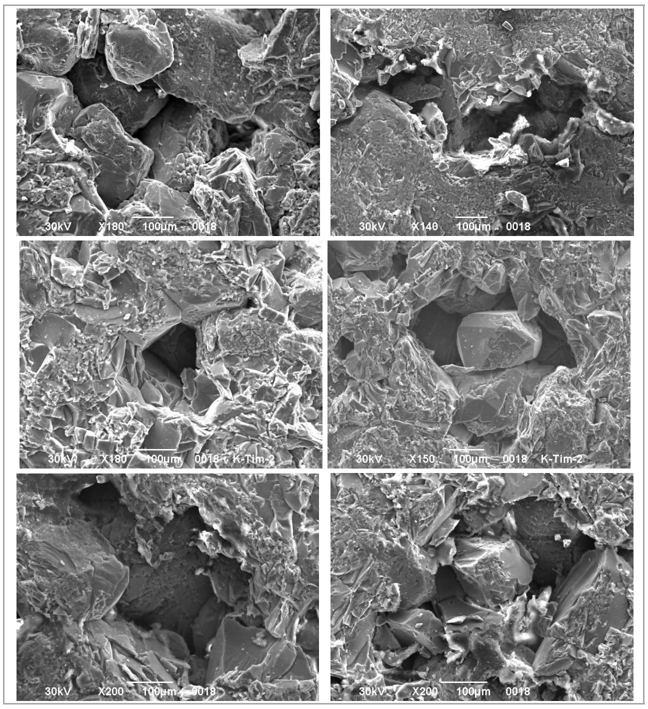

Fig. 1. Typical microcavities in interbeds of Visean sandstones with high reservoir properties

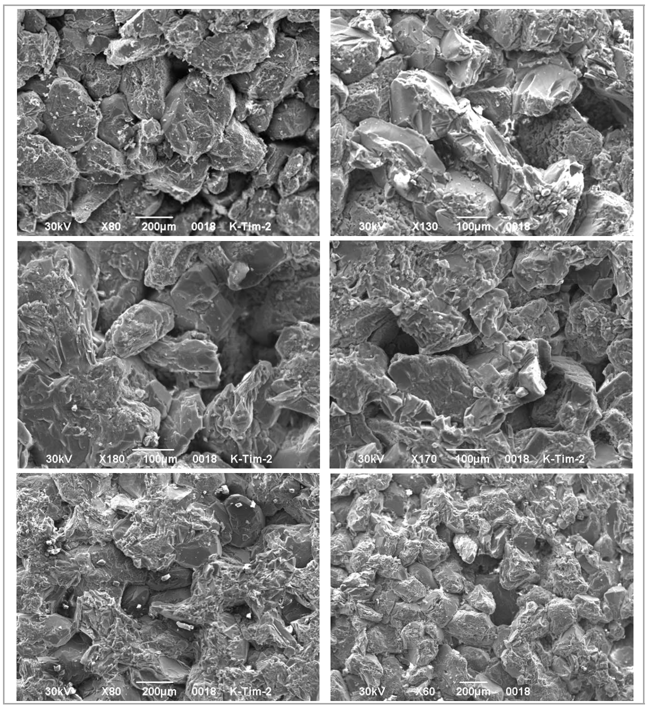

Fig. 2. Intergranular porosity of Visean sandstones interbeds with high reservoir properties

Further, the structural void space features of individual microcavities were studied using electron microscopic images obtained at a magnification of up to 200X. The largest microcavities are elongated in shape (some are more than 1 mm in the long axis and up to 0.2 mm in the short axis). They often have branches in the form of fractured zones, which continue their structure and create a pinch-out. Other microcavities have various shapes (isometric, irregular, rectangular, etc.) (Fig. 1).

Usually, the voids of microcavities are filled with quartz grains, but they leave a observable volume of pore space. Notably, the cementing material is rarely present and in small quantities.

Intergranular voids. A significant part of the void space in sandstones is made up of intergranular pores. Substantial differences are revealed in the nature of intergranular porosity for sandstones that vary in porosity and permeability values, allowing them to be divided into two groups.

The first group includes sandstone samples with high porosity and permeability properties, i.e., those that also show the presence of microcavities. Surface scanning of such samples during the study of intergranular voids was conducted using low microscope magnifications (from 60X to 180X). The intergranular pores in them have a localized distribution, with void sizes typically ranging from 0.3 to 0.1 mm. The shapes of intergranular voids can be triangular, angular, rectangular, slit-like, etc. (Fig. 2). Often, adjacent pores are connected with each other, forming extended channels of a tortuous shape.

The intergranular porosity is the result of a particular structure formation of the clastic sand-gravel sediment at the sedimentogenesis stage, mainly composed of well-sorted quartz grains by size. During the diagenesis and catagenesis stages, the rock is formed, maintaining the same clastic framework. In the grain size distribution of such sandstone, a single mode is distinguished on the histogram. The corresponding unimodal histogram of the grain size sandstone composition is presented in Fig. 3.

In natural conditions, such composition is a feauture of coarse- and medium-grained sandstones, formed during sedimentogenesis in environments with active water flow dynamics (river channel and coastal marine facies). Active dynamics of the water environment throughout the entire sediment accumulation period is essential for achieving a high degree of grain size sorting. The intergranular space in such sandstones remains virtually unfilled. The sizes of intergranular pores are a function of the grain sizes of the clastic framework and their packing density.

When studying the structures under an electron microscope, a relatively dense packing of clastic framework grains (mainly quartz grains, and less frequently feldspar) is observed, with a minimal amount of filler particles between them. As a result, the sandstone structure is characterized by a wide development of intergranular pores, which often contact each other, forming long, winding channels. It is also notable that there is a small amount of cementing material, mainly clay and other new substances.

The model applied to this type of reservoir features is a unimodal grain size distribution with a peak (in this case, 0.4–0.315 mm) of the clastic framework, which determines the size of intergranular pores to be approximately 0.2 mm. The variations in permeability for such a reservoir are largely influenced by the packing density of the clastic grains. The density is mainly controlled by their shape, which can be accurately determined under an electron microscope. For these reservoirs, the grain shape is close to isometric, which accounts for the significant volume of intergranular void space.

Moreover, the shape of quartz grains in the clastic framework also characterizes their roundness, which indicates the facies conditions of sedimentation. Among the quartz grains in the reservoirs of this group, semiangular grains usually dominate, corresponding to the facies settings of river basins. However, some grains are poorly rounded, and their surfaces retain smooth fragments of primary crystal faces. This feature indicates the presence of grains in the rock that come from different sources of clastic material, which can be used for detailed paleogeographic reconstructions.

In a subordinate quantity, feldspar grains are also present in the clastic framework of the sandstones from the studied deposits. Unlike quartz grains, feldspar grains are a subject to significant impacts from catagenetic processes, such as kaolinitization. The feature, with assessment of the processes intensity, can be used to determine the stage of catagenesis, which is an important diagnostic indicator for the potential oil and gas content of the geological section.

The second group includes rock cores for which, along with grains of the clastic framework, the presence of clastic filler particles is also identified. The filler particles occupy the voids between the grains of the clastic framework. Their occurrence is attributed to changes in the facies conditions of sediment accumulation, alternating between high and low flow rates. Filling the intergranular pores with filler grains leads to a noticeable decrease in the reservoir properties.

To account for the features of the granulometric composition of such reservoirs, a model with a bimodal histogram of particle size distribution can be used (Fig. 4). In such a histogram, the first peak corresponds to the grains of the clastic framework (usually 0.4–0.315 mm or 0.315–0.25 mm), while the second one corresponds to the filler particles (usually 0.16–0.125 mm or 0.125–0.1 mm).

The spatial arrangement of grains in reservoirs of this type is clearly visible in scanning electron microscope images (Fig. 5). The filler is mainly composed of quartz grains, but there is also a significant presence of feldspar grains. Morphological features of the filler grains include a weaker degree of rounding, an abundance of acute-angled individuals, and the presence of grains with perfect crystallographic faceting.

A separate group includes a siltstone sample, which differs from sandstones in all lithological features. The most important feature of this rock type, observed under the electron microscope, is the absence of a clastic framework. The reason for that may be an insufficient number of large quartz grains (Fig. 6).

The particle size distribution model of this type is a bimodal histogram of fragment size distribution, with a prevalence of the silt-sized mode (Fig. 7). For this model, typical large intergranular pores found in sandstones are absent; however, small intergranular voids up to 10 μm in size are quite common. The formation of the structure is possible only under conditions of an unstable facies environment of sedimentation, with extremely limited involvement of periods characterized by active water flow dynamics.

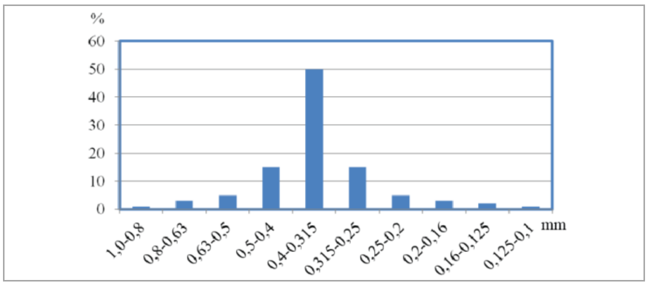

Fig. 3. Model of a histogram with a unimodal grain size distribution for sandstones with high reservoir properties

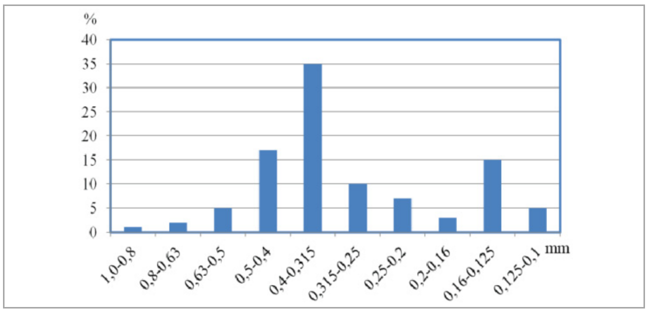

Fig. 4. Model of histogram with unimodal grain size distribution for the sandstone with lower reservoir properties

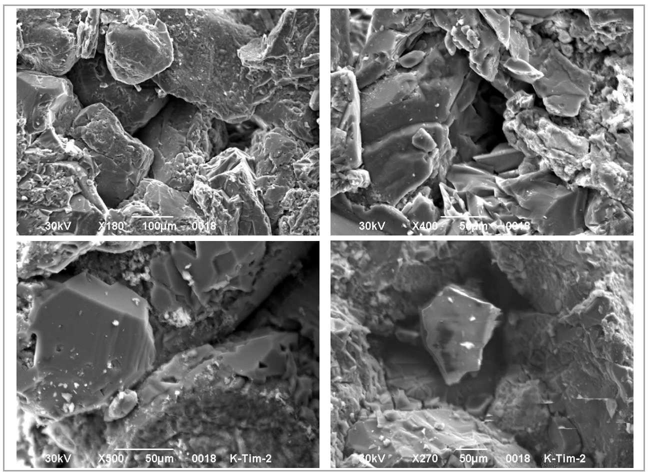

Fig. 5. Filler grains in intergranular voids of sandstones

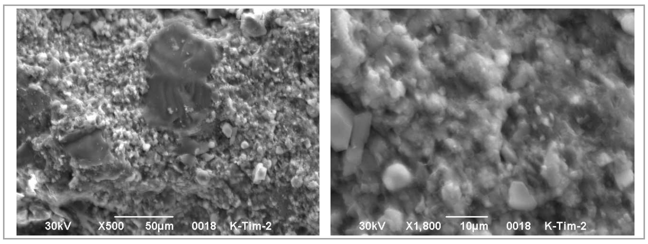

Fig. 6. Fragments of siltstone structure

Study of cementing material by scanning electron microscopy methods

The structural features of the cementing material in reservoirs are considered in numerous works [20, 21]. Particularly, in international practice kaolinite cement is emphasized due to the specifics of its transformation at different stages of catagenesis [22, 23].

The cementing material in the Visean sandstones of the Solikamsk region is found to have a local

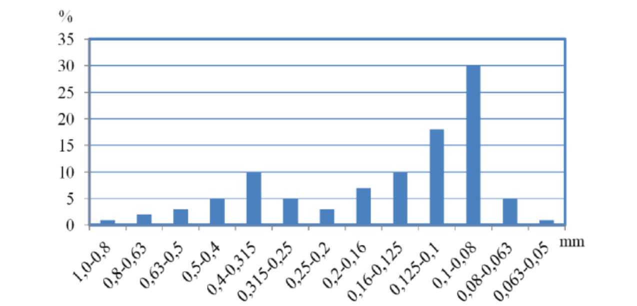

Fig. 7. Model of the grain size distribution histogram for silt fraction

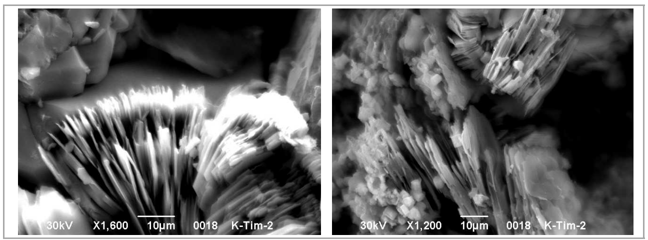

Fig. 8. Perfect kaolinite aggregates of "book house" type in sandstone with high reservoir properties

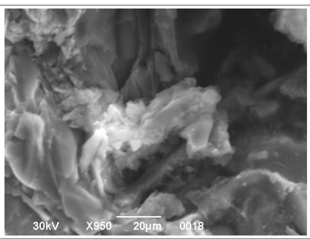

Fig. 9. Kaolinitized feldspar grain

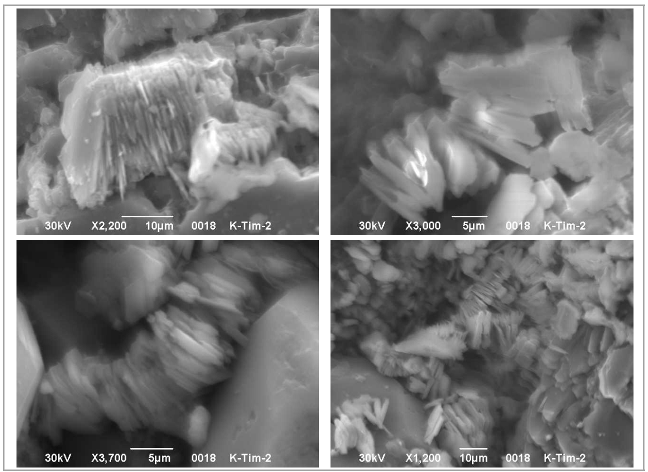

Fig. 10. "Book-house" type kaolinite structures in sandstone with low reservoir properties

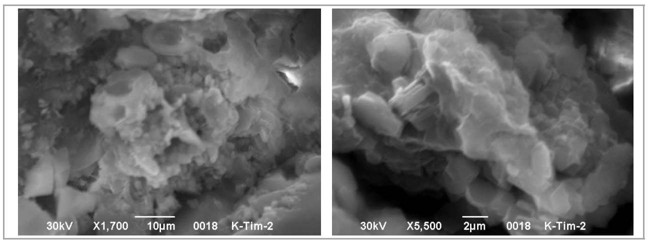

Fig. 11. Kaolinite aggregates in the cementing material of siltstone

distribution. It has been identified only in certain intergranular voids and is mainly represented by aggregates of kaolinite particles. In sandstones with high porosity and permeability, kaolinite forms characteristic microstructures known as "book-house" types, which vary significantly in terms of their perfection. The nature of the kaolinite aggregates in the studied sandstones shows a clear division into the same two groups that were identified during the analysis of the granulometric composition.

The first group is characterized by the most perfect structures of kaolinite aggregates in the "book-house" type. A distinctive feature of the structures is the presence of columns (packs) made up of plate-like newly formed kaolinite particles in a subparallel orientation (Fig. 8). The most perfect kaolinite structures are found only in select samples of sandstones, which show the highest reservoir properties.

The initial substance for their formation was grains of feldspar within the clastic framework or filler, which are characterized by their instability under the influence of catagenetic factors (such as groundwater, sulfur, organic matter, etc.). An example is provided by a partially kaolinitized feldspar grain discovered within the filler (Fig. 9).

The second group of "book-house" structures is characterized by a less perfect structure, which appears in smaller column sizes, irregularities in their composition, and other features (Fig. 10). Such structures are typical for sandstones characterized by lower values of reservoir properties.

In the siltstone sample, "book-house" structures were not observed. Notably, the cementing material in this case is widespread and is also kaolinite in composition (Fig. 11). However, here, kaolinite forms dense amorphous aggregates in which there are only a small number of inter-aggregate and intra-aggregate voids, measuring up to 5 μm.

A certain correlation between the perfection degree of "book-house" type structures and the productivity of reservoirs is due to the influence of this factor on porosity and permeability coefficients of the reservoirs. The reason is the increase in the volume of microporous space in the form of inter-aggregate voids between the newly formed plates of kaolinite. These plates originated during the catagenesis stage as a result of the primary (hypergenic) particles coarsening. Additionally, a favorable factor contributing to the enhanced oil-bearing capacity of the formations is the the kaolinite plates hydrophobicity.

A reliable indicator of the monomineralic kaolinite composition of the cementing material in sandstones with "book house" type aggregates is provided by microprobe analysis data (Table 1). Among the accompanying elements, the presence of calcium stands out, which is a consequence of the associated carbonatization process.

The formation of "book-house" type structures occurs during the mesocatagenesis stage, which is most favorable for the potential productivity of reservoirs. These structures are extremely sensitive to catagenesis factors (such as pH of the environment, associated superimposed processes, etc.), allowing them to be used for more detailed analysis of rock changes during catagenesis, assessment of micro-porosity features, and overall oil content forecast of rocks.

Table 1

Chemical composition of "book-house" kaolinite aggregates, wt. %

Oxide	Aggregate number									
	1	2	3	4	5	6	7	8	9	10
SiO_2	47.70	52.15	46.04	54.46	57.01	49.21	48.13	43.49	42.13	47.86
TiO_2	0.28	0.24	–	0.10	0.53	0.08	–	0.06	–	0.05
Al_2O_3	36.97	33.13	38.89	30.77	26.99	35.59	36.33	42.07	37.40	32.76
CaO	0.12	0.23	–	0.19	0.62	–	1.44	0.06	2.15	0.02
MgO	0.16	–	–	–	0.15	0.19	0.06	–	0.08	–
FeO*	0.14	0.10	0.04	0.42	0.37	0.53	–	0.13	0.03	0.21
K_2O	0.30	0.16	0.07	0.07	0.34	0.39	0.04	0.11	0.11	0.09
Na_2O	–	–	–	–	–	–	–	0.08	0.09	–

Table 2

Chemical composition of oxide mineral formations, wt. %

Oxide	Analysis number						
	1	2	3	4	5	6	7
V_2O_5	–	2.52	–	–	–	–	–
P_2O_5	–	–	36.44	34.75	–	–	–
SO_3	–	–	1.12	3.98	0.22	2.65	1.29
SiO_2	9.77	3.30	14.45	14.75	3.60	–	12.37
TiO_2	89.41	90.50	–	–	0.95	3.74	0.19
Al_2O_3	0.64	0.55	2.27	1.67	1.10	–	6.24
CaO	0.03	0.20	39.14	37.21	0.43	0.38	4.77
MgO	–	0.08	–	–	–	–	8.07
FeO*	0.13	2.76	0.09	1.89	–	–	65.79
MnO	–	0.06	–	–	–	–	–
BaO	–	–	–	–	0.35	0.94	–
CuO	–	–	–	–	38.38	29.66	–
K_2O	0.03	0.03	–	–	–	–	0.99
Na_2O	–	–	0.49	0.32	–	–	0.30
F	–	–	5.96	5.43	–	–	–
Cl	–	–	0.04	–	–	–	–

Note: leucoxene: *1* – Unvinskoye deposit, *2* – Siberian deposit (see Fig. 12, *1*); apatite: *3, 4* – Siberian deposit (see Fig. 12, *3, 4*); malachite: *5, 6* – Siberian deposit (see Fig. 12, *5, 6*), *7* – iron mineralization, Unvinskoye deposit.

Table 3

Chemical composition of pyrite formations, wt. %

Element	Field	
	Unvinskoe	Siberian
Fe	43.94	45.33
S	55.05	54.31
Ti	0.79	–
Co	0.22	0.18
As	–	0.18

Study of superimposed mineral formation processes using scanning electron microscopy methods

Post-sedimentation changes in reservoirs significantly affect the potential oil and gas content of rocks. Often, their porosity and permeability are substantially modified during diagenesis and catagenesis stages due to the filling of void space with newly formed substances. For instance, the process of intensive natural cementation has led to a significant reduction in the reservoir properties for the core samples of siltstone. In cores composed of sandstones, the following processes of secondary mineral formation have been recorded using microprobe analysis: leucoxenization, pyritization, phosphatization, ferruginization, and copper mineralization (Fig. 12, Tables 2 and 3).

The leucoxenization process results in the formation of dense aggregates larger than 0.1 mm in size, made of thin-layered leucoxene, which fill specific voids between the grains of the clastic framework. The process of pyritization leads to the formation of well-formed tabular pyrite crystals packets in parallel orientation, as well as fine-grained aggregates. The process of phosphatization has also been noted leading to the formation of apatite crystal aggregates of plate-like or columnar forms. Finally, superimposed copper mineralization has been discovered as deposits of malachite aggregates.

The role of the described superimposed processes is to establish the participation of the reservoir organic matter in secondary mineral formation as a specific factor of catagenesis. Thus, they can serve as an additional indicator of their oil content. It is important to consider that all chemical elements involved in the superimposed processes (Fe, Ti, P, S, Cu, and V) are typical impurities found in organic matter.

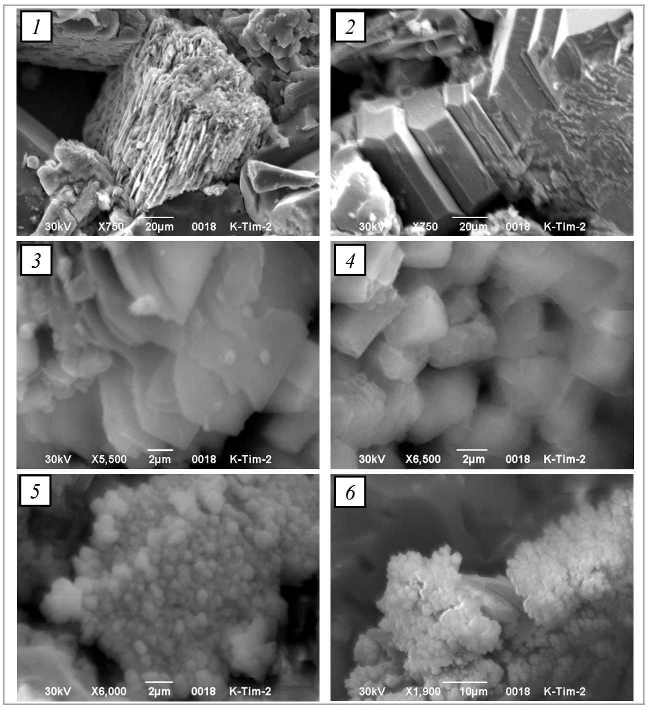

Fig. 12. Secondary formations in oil-bearing sandstones: *1* – leucoxene; *2* – pyrite; *3, 4* – apatite; *5, 6* – malachite

At the same time, the transformation of the void space in the reservoirs as a result of the superimposed mineral formation processes is not so significant due to their localized manifestation. It amounts to the partial filling of certain intergranular voids and the generation of newly formed intergranular pores up to 5 µm in size.

Conclusion

Electron microscopy methods make it possible to identify and study in detail the lithological features of terrigenous productive strata. In general, the reservoir properties are largely determined by the structural features of the sediment, which are laid down at the stage of sedimentogenesis and are influenced by the facies sedimentation environment. The main role in this process is played by a clastic framework formation, which usually consists of quartz grains. Important components of the terrigenous reservoir are the mineral grains of the filler and the cementing material composition. The superimposed processes of diagenesis and catagenesis especially influence the void space, significantly the mineral composition and microstructure of the cementing material. In general, the use of electron microscopy methods allows us to identify different rocks features that affect their porosity, permeability and potential oil content.

In the studied sand reservoirs of the Solikamsk depression, two groups have been identified, for which a regular relationship between productivity and granulometric composition features, void space, the composition and structure of cement has been established.

The first group of reservoirs is characterized by good sorting and a dense packing of quartz clastic framework grains, a small amount of filler particles, and a cementing material represented by kaolinite with a "book-house" type structure.

The second group of reservoirs with lower permeability is characterized by weak sorting of grains due to the presence of a significant amount of filler particles in the clastic framework, which significantly reduces their reservoir properties. An increased proportion of cementing material also contributes to a decrease in porosity and permeability. A characteristic indicator of such a reservoir is a less perfect kaolinite microstructure of the "book-house" type.

The following lithological features were established for the studied siltstone core: multi-grained granulometric composition, absence of grain sorting by size, unstructured kaolinite cement with absence of “book-house” type structures.

Such significant lithological differences among sandstones of the same geological age determine their oil potential. They are conditioned by the important role of the facies environment of sedimentation and subsequent processes of rock transformation at the stages of diagenesis and catagenesis. A crucial indicator of these processes is the microstructure of the clayey material.

The lithological indicator features studied by electron microscopy methods and described in the article can be recommended for wider application in petroleum geology.

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