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# DETAILED STUDY OF HYDRAULIC FRACTURING OF KASHIRO-VEREY ROCKS BY ELECTRON MICROSCOPY

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***Summary.*** The fracturing of carbonate rocks of the Kashiro-Verey formation in the Volga-Urals region is studied using X-ray tomography and electron microscopy methods. The lithological and structural features of the formation consisting of four lithotypes are described. The lithotypes differ markedly in the degree of clay material, the nature of the void space, and reservoir properties. Two lithotypes are weakly permeable. Experimental modeling of the effect of proppant hydraulic fracturing technology on core samples is applied.

It is established that loading affects the interlayers differently depending on their structure and material composition, and the presence of clay and the nature of porosity have the greatest influence. The microcracks of hydraulic fracturing under loading of samples develop to the maximum extent in the densest rocks, in which extended cracks with an opening of about 100 μm or more are formed. In turn, in the permeable part of the lithotypes, extended, usually narrow (thickness less than 20 μm) rectilinear cracks are also formed, oriented in one direction and connecting neighboring microcaverns and pores. Their strict orientation in space reflects the direction of the load on the samples, but may also be partly due to the hidden microlayering of the rocks.

The conclusion is made about the possibility of forming through oil migration channels in the reservoir. The results of the experimental study will lead to increasing of the using efficiency of the hydraulic fracturing technology in the operation of the Kashiro-Vereya productive reservoirs.

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## 1. Introduction

In the last decade, one of the most promising in increasing oil production of operational facilities in the Volga-Urals oil and gas province are oil deposits of Kashiro-Verey age. The most active development of these operational facilities is observed in the territory of the Perm Region.

The initial recoverable reserves for the Kashiro-Verey deposits of the Perm Region exceed 130 million tons (as of 01.01.2022) with an average reserve production of about 30%. The actual drilling capacity of facilities relative to the project fund is on average 50% with an average water content of 30% of well production (the range from 5 to 74%). Thus, Kashiro-Verey objects as a whole are characterized by high residual oil reserves with the low water content of well production with the potential to increase the well stock. The use of modern technologies for this territory makes it possible to rapidly introduce Kashiro-Verey deposits of "old" fields into active, profitable development, up to a 10-fold increase in oil production (Воеводкин, Антонов, 2021).

The Kashiro-Verey deposits of the Volga-Urals are an interlayer of mudstones and carbonate rocks,

and the carbonate stratum is characterized by varying degrees of clay material. The most clay-free interlayers are distinguished as collectors, but the total thicknesses of this type of rocks, as a rule, do not exceed 2-6 m. As the clay material increases, the reservoir properties of the layers naturally deteriorate, and significant difficulties accompany the production of reserves from them. They can be overcome with the use of various technologies. However, the use of standard technologies such as hydrochloric acid treatment (Novikov, Martyushev, 2020; Novikov, 2021) and radial opening of the reservoir (Chi et al., 2015; Galkin et al., 2019) is accompanied by a rapid decline in oil production and is characterized by low profitability.

The most effective technology for the development of the above deposits for the territory of the Perm Region in recent decades is considered to be proppant hydraulic fracturing (Raspopov et al., 2019; Votinov et al., 2021; Галкин и др., 2021), as a result of which the rock breaks along the planes of minimum strength. This technology has long been used in world practice in the development of hard-to-recover oil reserves (Penny et al., 1985; Усачев, 1986; Каневская, 1998). In many countries of the world, including Russia, significant efforts have been made to introduce new developments in this area (Economides, Nolte, 2006; Salimov et al., 2013). In recent years the technology has become increasingly widespread due to the depletion of oil reserves in many oil-producing countries (Яркеева, Хазиев, 2018; Wright, Muma, 2018). Reviews of the application of hydraulic fracturing technology in individual oil fields are given in some works (Здольник и др., 2016; Топал и др., 2018).

Hydraulic fracturing cracks are fixed after pressure is removed by proppant and become reliable and long-term oil conductors. The ability of the layer to conduct liquid through the created crack and deliver it to the wellbore is determined by the concept of dimensionless crack conductivity (Smith, Shlyapobersky, 2000). The geometry of hydraulic fracturing cracks is determined by their half-length, height, and width (Muther et al., 2020) and significantly affects the effectiveness of this technology.

The effectiveness of propant hydraulic fracturing technology largely depends on the characteristics of cracks (the number of cracks, their extent, shape, orientation in the rock, the degree of disclosure, etc.). In turn, these parameters depend on the material composition and structure of the rock. Of particular importance is the real idea of the natural fracturing of rocks (Насыбуллин и др., 2010). In addition, it is necessary to take into account the peculiarities of the structure of the oil reservoir (Aliyeva, Mustafayev, 2021; Gurbanov et al., 2019, 2021a,b).

However, methods for predicting hydraulic fracturing using the developed algorithms and computer programs have not yet been developed. Nevertheless, at this stage, such a problem can be solved with the use of appropriate laboratory and field experiments with core samples. Examples of such studies are given in a number of papers (Laubach et al., 2004; Fjaer, 2008; Ibatullin et al., 2009).

## 2. Object and methods of research

Fracturing, both natural and after hydraulic fracturing, can be primarily studied at the macrolevel. Averaged data on the fracturing of the working perforation intervals (thickness – the first tens of meters) can be obtained from the pressure recovery curve (KVD). According to the Warren-Root model, the presence of two parallel lines on the KVD and a characteristic inflection between them are interpreted as a manifestation of fracturing (Cherepanov, 2015; Martyushev et al., 2019; Martyushev, Yurikov, 2021). In the paper (Votinov et al., 2021), based on the processing of KVD for the Kashiro-Verey object of the Moskudyinsky field, a scheme of natural fracturing was constructed, while the crack opening was estimated as low (less than 10 μm).

Fracturing at the level of individual interlayers (thickness from 0.2 to 3 m) can be studied according to the data of geophysical studies of wells. The complex of natural gamma (GC) and neutron-neutron logging (NNA-t) is the main one for Kashiro-Verey deposits in determining reservoir properties. The GC method is characterized by the clogging of layers with a clear separation of limestone interlayers ($\Delta I_{gc}$<0.3 d.e.) and mudstones ($\Delta I_{gc}$>0.5 d.e.). The minimum values of NNA-t are fixed on the most porous limestones, the maximum – the densest ones in the carbonate section.

The analysis of the core material made it possible to identify four lithotypes of rocks for Kashira-Verey limestones that differ in structural features (Galkin et al., 2021). The intervals of limestone without clay ($\Delta I_{gc}$<0,03; $\Delta I_{nnk}$ <0,17) are treated to porous-cavernous lithotype 1. Lithotype 2 is represented by alternating 2-10 cm porous and dense differences of the rocks in the ratio 76% to 24% ($\Delta I_{gc}$ from 0.07 to 0.08; $\Delta I_{nnk}$ from 0.24 to 0.26). The dense layers are dominated (86%) in lithotype 3 ($\Delta I_{gc}$ from 0.07 to 0.19; $\Delta I_{nnk}$ from 0.30 to 0.37). Porous areas in these rocks are represented as inclusions, and the presence of fracturing has been established for them. The increased $\Delta I_{gc}$ figures (0.17-0.31) indicate the predominant clayey composition of limestones for lithotype 4, and the range of $\Delta I_{nnk}$ (0.45-0.49) indicates their strong compaction. At the same time, even for the densest lithotype 4, non-zero porosity (Kp = 6%) is established according to the core data, which shows the proximity of the intervals without no signs of oil to the collectors.

Fracturing at the microlevel can be assessed based on the results of core studies. To solve this problem, both standard core research methods and special ones can be used. The objective of this work is a detailed study of the processes of hydraulic fracturing using scanning electron microscopy methods (Quattro-C, ThermoScientific, USA). The operating regime for the microscope is the following: voltage 20 kV, the working distance is 10 mm, the current is 1 nA. At the same time, the entire surface of the samples is scanned at magnifications from 100 to 30,000 times (the resolution of the microscope at the last magnification is 0.1 μm).

The electron microscopy results are supplemented with data from X-ray tomography of the core, which allow visualizing large pores and cracks with relatively high disclosure in the sample volume (Efimov et al., 2015; Pakzad et al., 2018). For this task, a Nikon Metrology 125x180 XT scanning unit is used, which makes it possible to visualize cracks with an opening of 20-30 μm or more on a standard core.

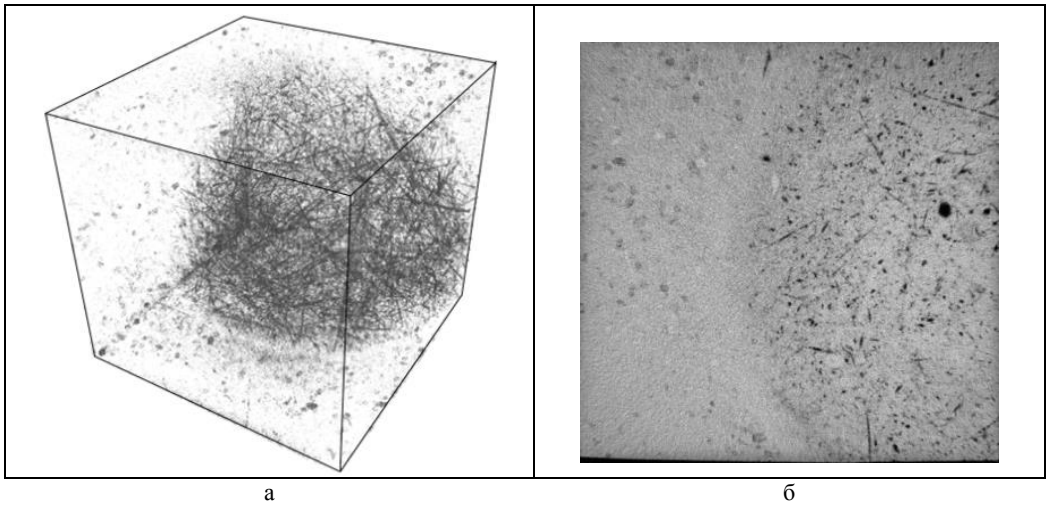
As shown above, Kashiro-Verey formations are characterized by the presence of weakly fractured zones, which in their natural state do not significantly affect the processes of fluid filtration. At the same time, when implementing hydraulic fracturing technology, microcrack systems can be significantly activated. To assess the scale of this process, cubic core samples with a side of 40 mm are made for all four lithotypes of rocks, and experiments on their multiaxial loading are carried out (Karev et al., 2016, 2017).

At the beginning of each experiment cycle, the same pressure on the sample was created from all sides. Compression in experiments for lithotypes 1, 2, and 3 was ranged from 10 to 20 MPa, and did not exceed 10 MPa for lithotype 4. Taking into account the assessment of the effective pressure in the formation conditions of Kashiro-Verey deposits of the order of 12 MPa, the pressure of extremely rigid compression created in experiments is comparable to real reservoir conditions. Then, with the support of constant pressure along two axes (on four faces of the cubic sample), loading along the third axis (on the two remaining faces) was created up to the values of the maximum axial load.

The maximum values of the axial load on the samples in the experiments were, respectively, 40 MPa – for lithotype 1; 80 MPa – for lithotype 2; 100 MPa – for lithotype 3; 140 MPa – for lithotype 4. At the same time, the limit of elastic deformation in the experiments was not exceeded, but the samples could be re-compacted relative to their natural state. In general, the experiments carried out on the cores could be compared with the conditions of hydraulic fracturing in the formation.

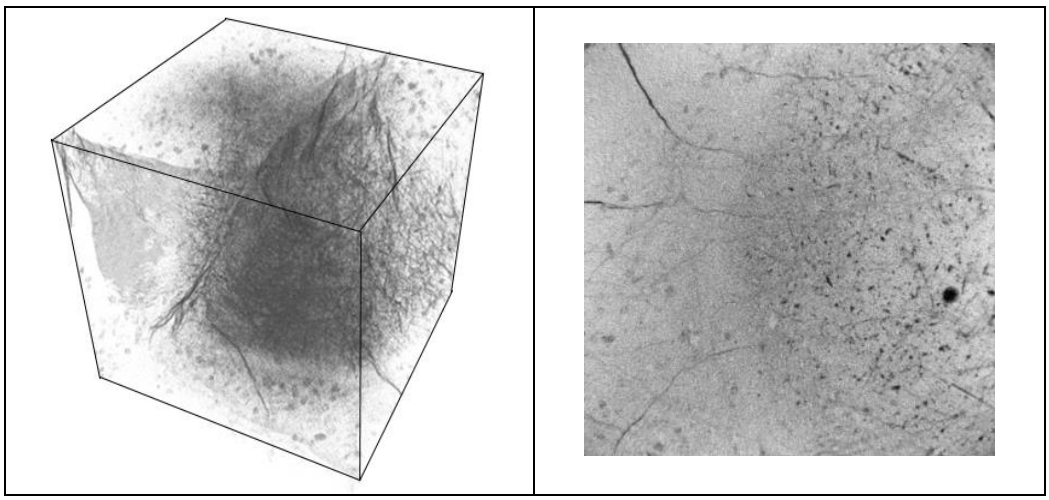
The analysis of the impact of the load on the formation of fracturing are carried out by comparing the state of the cores before and after exposure. In this case, first of all, X-ray tomography data are used. The method of X-ray tomography, in addition to studying the pore space, allows you to visualize the presence of relatively open cracks in the sample volume. At the same time, the capacitive space on the tomograms is highlighted in black, areas with increased X-ray density are white.

X-ray tomography shows that as a result of experiments in lithotypes 2, 3, and 4, intensive formation of hydraulic fracturing cracks occur. Figures 1 and 2 show tomograms of cores for the most problematic lithotype 3 (before and after multiaxial loading). The tomogram of the sample before the multiaxial loading shows a clear difference between the dense and porous parts of the rock, while no extended cracks have been established. Only short non-communicating cracks are visible on the cut in the porous part of the sample (Fig. 1). Existing cracks develop in the porous part, and intensive formation of extended cracks with an opening of about 100 μm occurs in the dense part after loading (Fig. 2).



а б

**Fig. 1**. Results of X-ray tomography in volume (a) and in section (b) before experiments on multiaxial loading of a lithotype 3 sample



а б

**Fig. 2**. Results of X-ray tomography in volume (a) and in section (b) after experiments on multiaxial loading of a lithotype 3 sample

### 3. Results of electron microscopy studies

Microcrackage in the initial samples, recorded on electron microscopic images, is poorly developed in lithotypes 1 and 2. Only short microcracks are present between some microcaverns and pores in them. Microcracks in lithotype 3 are more developed and more diverse. However, these microcracks are usually short (up to 5 μm), and the thickness does not exceed 0.5 μm. As to dense clayey lithotype 4 microcracking is practically absent (Fig. 3).

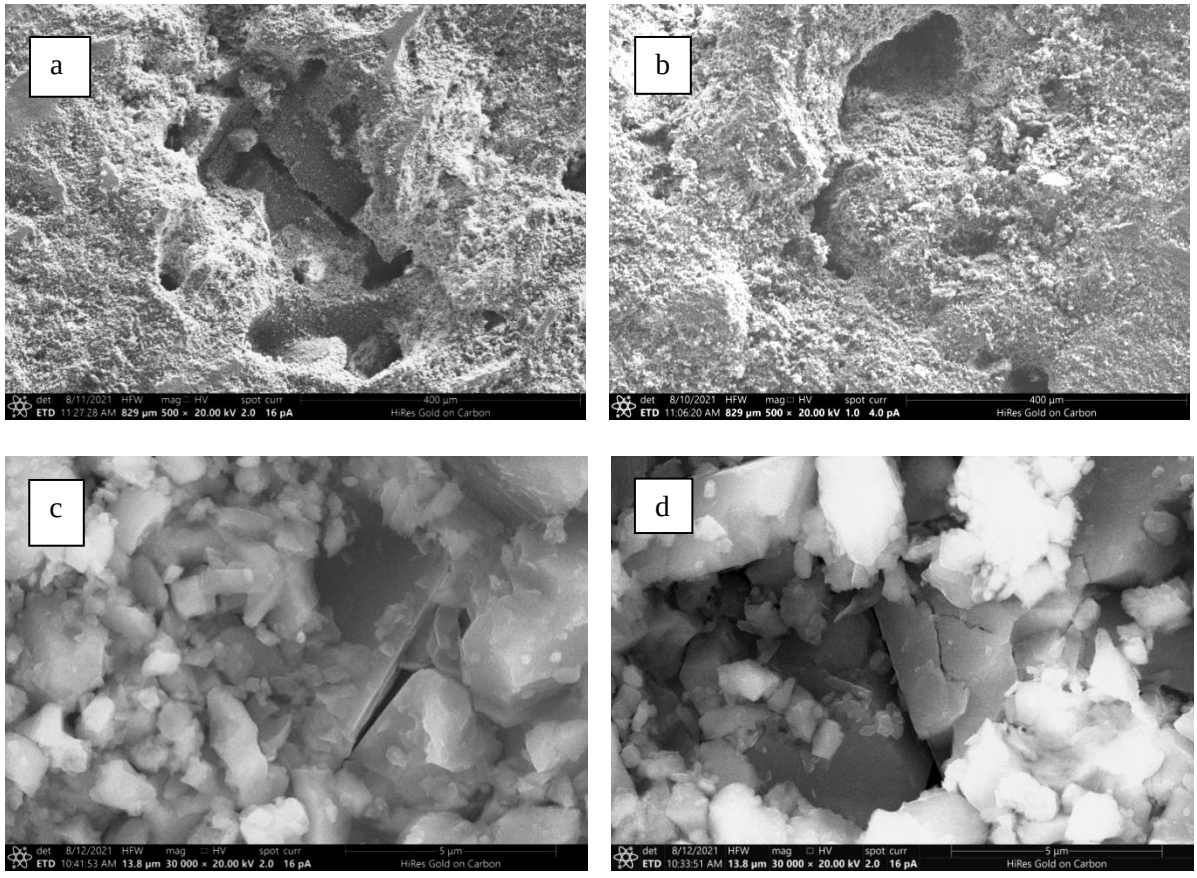



**Fig. 3**. Typical microcracks in samples before loading: a, b – lithotypes 1 and 2 (respectively); c, d – lithotype 3

After conducting experiments with samples of lithotypes, electron microscopy shows the intensive formation and a wide variety of hydraulic fracturing microcracks. At the same time, the hydraulic fracturing in ***lithotype 1*** is mainly represented by three types of microcracks:

1) a system of microcracks of few, relatively short (length up to 0.3 mm), sometimes strictly parallel and rectilinear, with a thickness of about 3-5 μm;

2) a system of numerous extended microcracks, about 10-20 μm microns thick, weakly branched, connecting microcaverns and pores (Fig. 4);

3) single short (up to 0.2 μm) cracks up to 50 μm thick.

Each of the above systems is confined to certain areas of the sample with a special structure of the hollow-porous space. Thus, pairs of close parallel microcracks are confined to zones of increased porosity. They are usually located in the areas between the converging microcaverns.

Systems of narrow extended microcracks with a thickness of several tens of micrometers appear under the influence of load in areas of reduced porosity and function as connecting channels between micropores. They are oriented in space in a certain direction sub-parallel to each other. Some of them cross the entire experimental sample.

Single short cracks differ from the previous ones with increased thickness (up to 50 μm) with extensions in some areas. They existed in the sample before loading, but were noticeably expanded and somewhat deformed.

Thus, the result of the impact of the load on the lithotype 1 rocks is mainly the formation of connecting channels between microcaverns and thereby an increase in the volume of contacting voids.

The result of the experiment for ***lithotype 2*** is the formation of several systems of hydraulic fracturing microcracks in zones with different permeability. In particular, two systems of microcracks are characteristic of *dense impermeable interlayers*: 1) microcracks extended in space with a thickness of up to 10-20 μm, 2) deep wide (up to 50 μm or more) cracks with branches.

Microcracks of the first type in the sample have a certain orientation (perpendicular to the direction of the load). Cracks can branch in some areas (Fig. 5). At the same time, cases have been noted when a certain section of the microcrack is healed by a newly formed carbonate substance. This fact can be interpreted as follows: previously existing microcracks were expanded and extended under the influence of load.

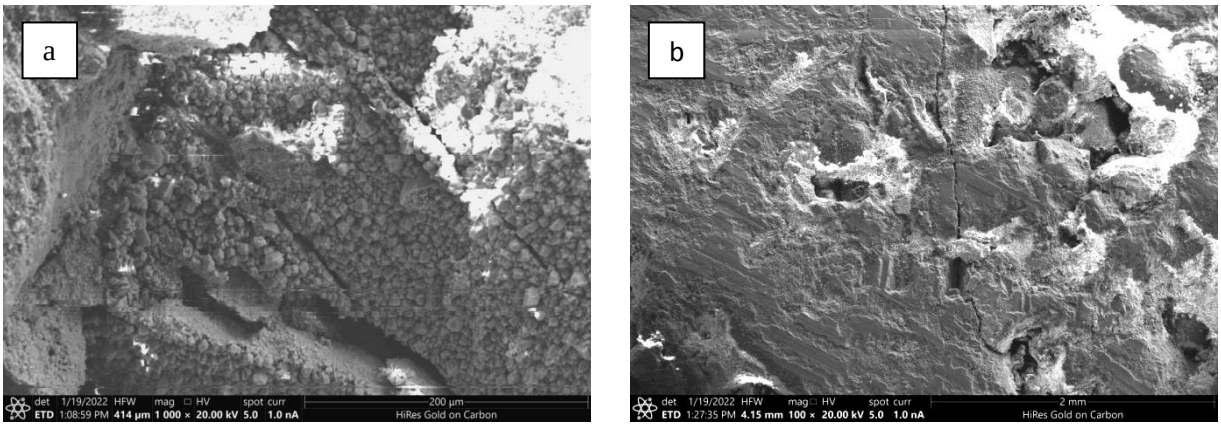



**Fig. 4**. Typical microcracks in lithotype 1: a – parallel, b – extended, connecting microcaverns and pores

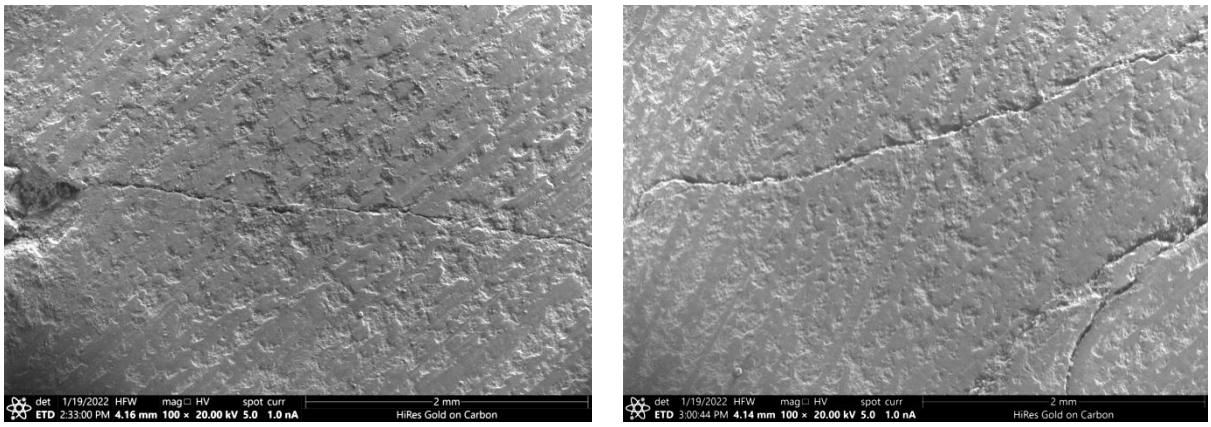


**Fig. 5**. Extended microcracks in the lithotype 2

Deep cracks with branching and local inflations up to 0.5 mm thick are less frequent and probably fix the areas of the most severely disturbed structure of the sample under the influence of load (Fig. 6).

*Porous permeable interlayers* are characterized by short or extended microcracks up to 10 μm wide, which usually connect microcaverns and large pores (as in lithotype 1). They have a certain orientation in the sample, coinciding with the general direction of cracks in dense interlayers, and probably reflect the direction of the load (Fig. 7).

***Lithotype 3*** is characterized by a wide variety of microcracks of different lengths, shapes and depths. Scanning of the sample surface after loading shows the presence of zones of increased cracking and the existence of several generations of microcracks, of which the subsequent ones intersect the earlier ones in the time of formation.

The appearance of deep extended microcracks with inflations up to 250 μm thick of different orientations is noted in *dense impermeable interlayers* (Fig. 8). The presence of three systems of hydraulic fracturing microcracks is noted for *porous zones*: 1) parallel narrow extended, 2) intersecting (Fig. 9), and 3) short connecting "channels" between microcaverns and pores.

The load had the most significant impact on the void space of the rock on ***lithotype 4***, represented by dense limestone. Extended microcracks (through the entire experimental sample) were formed in them after loading with a thickness of up to 100 μm in places, which are often grouped into parallel systems. An attention is drawn to the same orientation in space of all microcracks in the sample of this lithotype (perpendicular to the pressure) (Fig. 10).

## 4. Discussion of the results and conclusions

Experiments on multiaxial loading of samples, simulating the conditions of the technology of proppant hydraulic fracturing, have shown that complex fracturing microcracks are intensively formed in Kashiro-Verey rocks that are weakly fractured in their natural state during loading.

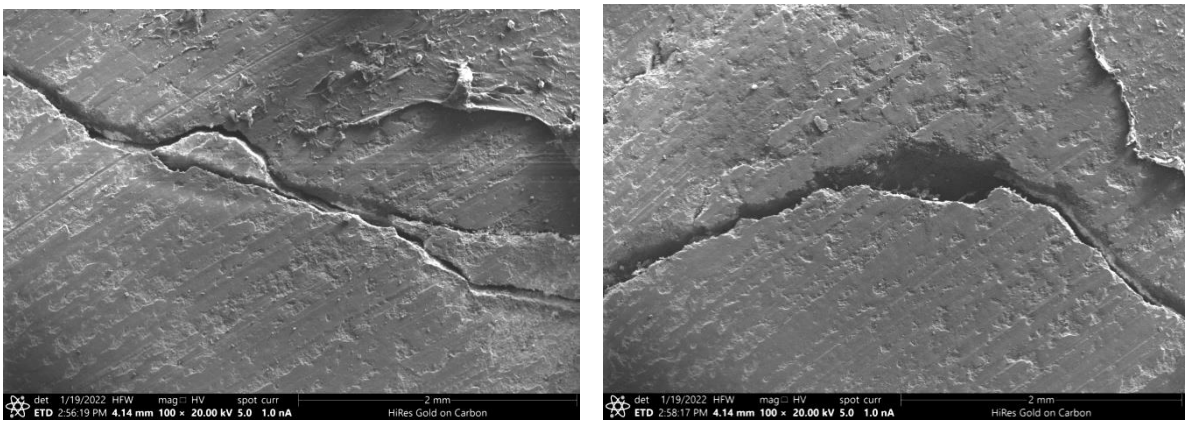



**Fig. 6**. Deep cracks with inflations and branching

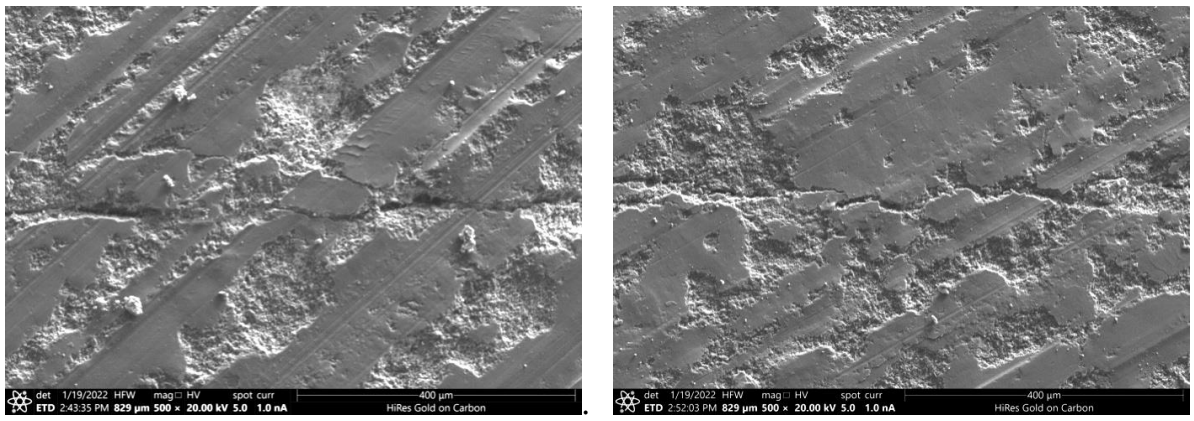



**Fig. 7**. Extended microcracks in areas of reduced porosity

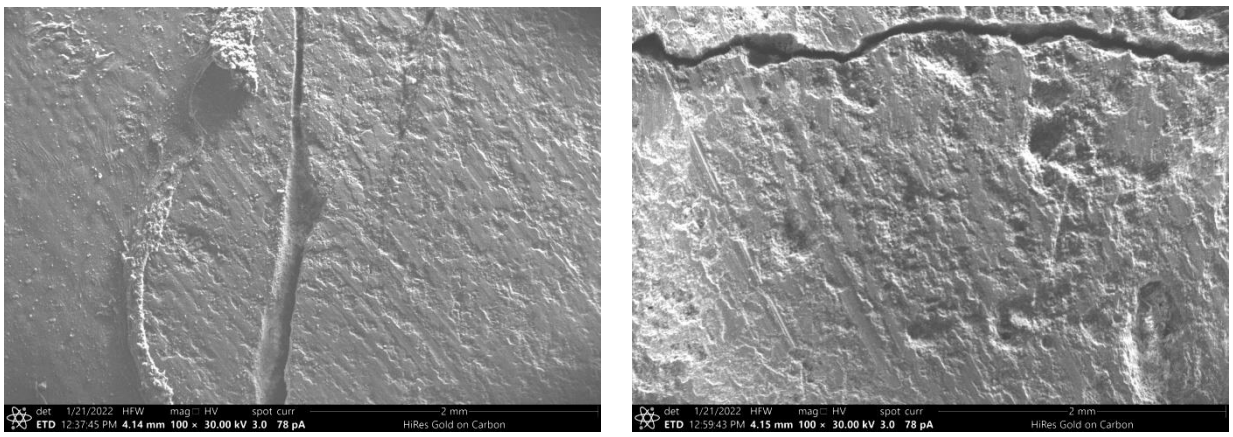


**Fig. 8**. System of deep extended cracks in dense interlayers of lithotype 3

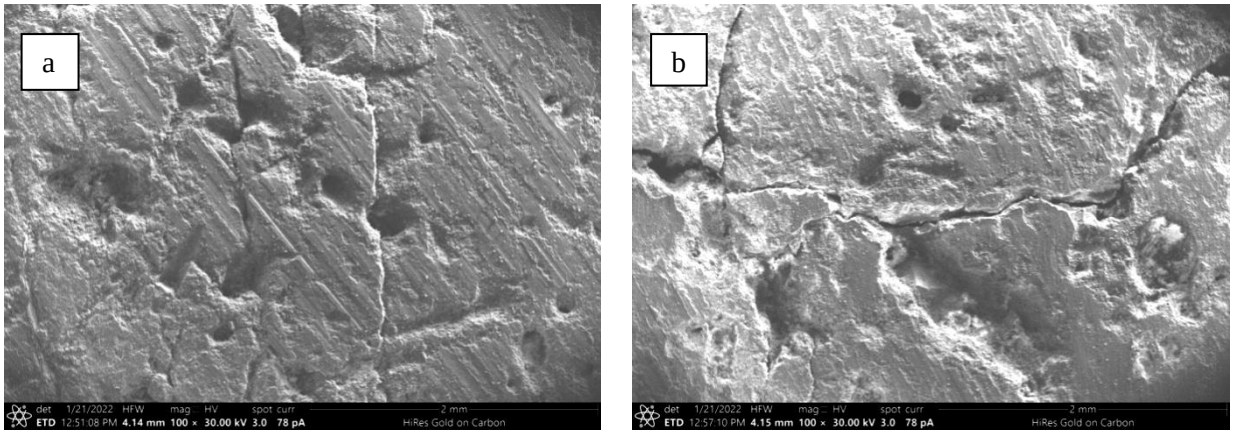



**Fig. 9**. Microcrack systems in porous interlayers of lithotype 3: a – parallel, b – intersecting

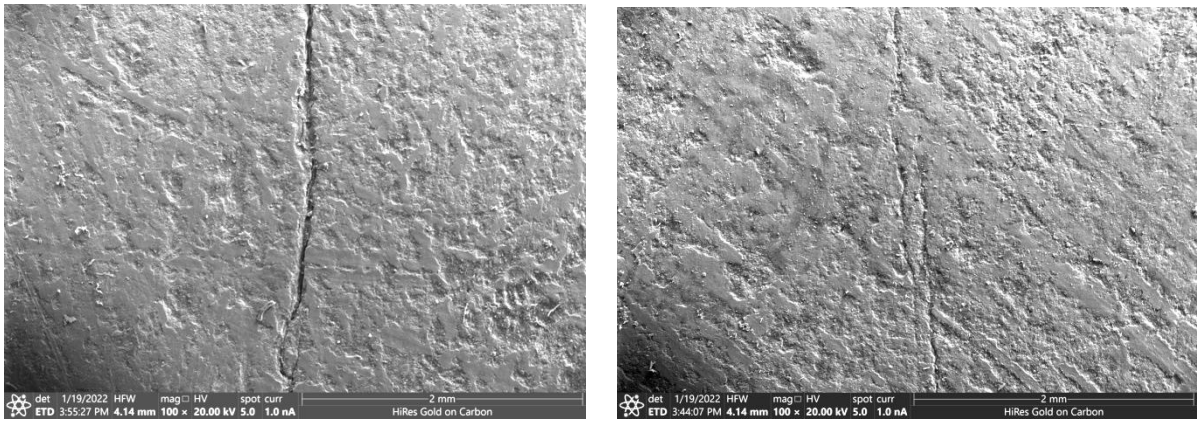


**Fig. 10**. Typical microcracks in lithotype 4

Combining the methods of X-ray tomography and scanning electron microscopy of the core allows us to establish the appearance of numerous systems of hydraulic fracturing microcracks of various types and lengths. At the same time, electron microscopy makes it possible to study in detail the morphology, orientation in the sample, thickness, frequency of occurrence, and other characteristic parameters of microcracks. The choice facilitates this for experimental studies of various lithotypes of rocks of the studied formation (from permeable to impermeable, and intermediate variants with a different ratio of those and other layers).

It is established that loading affects the interlayers differently depending on their structure and material composition, and the presence of clay and the nature of porosity have the greatest influence. The microcracks of hydraulic fracturing under loading of samples develop to the maximum extent in the densest rocks, in which extended cracks with an opening

of about 100 μm or more are formed. In turn, in the permeable part of the lithotypes, extended, usually narrow (thickness less than 20 μm) rectilinear cracks are also formed, oriented in one direction and connecting neighboring microcaverns and pores. Their strict orientation in space reflects the direction of the load on the samples, but may also be partly due to the hidden microlayering of the rock.

The experiments have confirmed that hydraulic fracturing technology contributes to the interconnection of permeable oil-saturated layers, thereby ensuring the complete extraction of oil from a complex reservoir. The experimental results suggest a high efficiency of using the technology of proppant hydraulic fracturing on Kashiro-Verey deposits with the formation of extended channels for oil filtration.

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## ДЕТАЛЬНОЕ ИЗУЧЕНИЕ ТРЕЩИНОВАТОСТИ ГИДРОРАЗРЫВА КАШИРО-ВЕРЕЙСКИХ ПОРОД МЕТОДОМ ЭЛЕКТРОННОЙ МИКРОСКОПИИ

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***Резюме.*** Методами рентгеновской томографии керна и сканирующей электронной микроскопии изучена трещиноватость карбонатных пород каширо-верейской свиты Волго-Уральской нефтегазоносной провинции. В пределах нефтеносной толщи описаны литологические и структурные особенности горных пород. Выделены четыре литотипа, которые различаются по степени глинистости материала, характеру пустотного пространства и коллекторским свойствам. Применено экспериментальное моделирование воздействия технологии пропантного гидроразрыва пласта на образцы керна. Детально изучено изменение трещиноватости горных пород под действием нагрузки на образцы, установлены различия данных процессов для каждого из рассмотренных литотипов. Установлено, что нагрузка по-разному влияет на прослойки в зависимости от их структуры и вещественного состава, причем наибольшее влияние оказывает наличие глины и характер пористости. Микротрещины гидроразрыва при нагружении образцов максимально развиваются в наиболее плотных породах, в которых образуются протяженные трещины с раскрытием около 100 мкм и более. В свою очередь, в проницаемой части литотипов также образуются протяженные, обычно узкие (толщиной менее 20 мкм) прямолинейные трещины, ориентированные в одном направлении и соединяющие соседние микрокаверны и поры. Их строгая ориентация в пространстве отражает направление нагрузки на образцы, но отчасти может быть обусловлена и скрытой микрослоистостью породы. В результате сделан вывод о формировании в процессе гидроразрыва дополнительных сквозных каналов фильтрации флюидов в пласте. Полученные результаты экспериментального исследования позволяют повысить эффективность применения технологии гидроразрыва пласта при эксплуатации каширо-верейских эксплуатационных объектов.

***Ключевые слова:*** *трещины гидроразрыва, карбонатный коллектор, керн, рентгеновская томография, электронная микроскопия, пропантный гидроразрыв пласта*

## ELEKTRON MİKROSKOP ÜSULU İLƏ KAŞİR-VEREYA SÜXURLARININ HİDRAVLİK PARÇALANMASININ ƏTRAFLI TƏDQİQİ

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***Xülasə.*** Volqa-Ural neft-qaz vilayətinin Kaşir-Vereya lay dəstəsinin karbonatlı süxurlarının qırılmaları kernlərin rentgen tomoqrafiyası və skaner elektron mikroskopiya üsulları ilə tədqiq edilmişdir. Süxurların litoloji və struktur xüsusiyyətləri neftli laylar daxi-

lində təsvir edilmişdir. Dörd litotip müəyyən edilmişdir ki, bunlar nümunələrin gilliliyin dərəcəsinə, məsamələrin təbiətinə və kollektorların xüsusiyyətlərinə görə fərqlənir. Proppant hidravlik parçalanmanın seçilmiş nümunələrə təsirinin eksperimental modelləşdirilməsi tətbiq edilmişdir. Nümunələrdə yükün təsiri nəticəsində qırılmaların fərqi ətraflı öyrənilmiş, nəzərdən keçirilən litotiplərin hər biri üçün bu proseslərdə xüsusiyyətlər müəyyən edilmişdir. Müəyyən edilmişdir ki, yüklənmə ara qatlarının strukturundan və maddə tərkibindən asılı olaraq müxtəlif təsirlər göstərir, gilin olması və məsaməliliyin xarakteri isə əlavə təsir göstərir. Nümunələrin yüklənməsi nəticəsində hidravlik qırılma mikroçatları, təxminən 100 mkm və ya daha çox açılışı olan uzanan çatların əmələ gəldiyi yüksək sıxlı süxurlarda maksimum inkişaf edir. Öz növbəsində, litotiplərin keçirici hissəsində davamlı, adətən dar (qalınlığı 20 mkm-dən az) düzxətli qırılmalar da əmələ gəlir, onlar bir istiqamətə yönəldilir və daxildəki mikrokavernaları və məsamələri birləşdirirlər. Qırılmaların istiqamətlənmiş oriyentasiyası təsir edən yükün istiqamətini əks etdirir, lakin bu qismən süxurun gizli mikrotəbəqələnməsi ilə əlaqədar ola bilər. Nəticədə belə qənaətə gəlindi ki, hidravlik parçalanma zamanı layda əlavə maye süzülmə kanalları əmələ gəlir. Eksperimental tədqiqatın əldə edilmiş nəticələri Kashiro-Vereya sənaye obyektlərinin istismarında hidravlik parçalanma texnologiyasının tətbiqinin səmərəliliyini artırmağa imkan verir.

***Açar sözlər**: hidravlik parçalanma, karbonatlı kollektor, kern, rentgen tomoqrafiyası, elektron mikroskopiya, proppant hidravlik parçalanma*