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The results of study of carbonate rock microstructural anisotropy obtained in electron microscopy and computer image analysis

Introduction

When water injection method is used to maintain reservoir pressure in the oil deposit extraction, the enhanced oil recovery depends to a considerable degree on the sweep completeness of oil-producing formation.

Sweep efficiency depends on many factors, in particular, on the features of technological oil field development and numerous factors of different scales, associated with the field's geological structure and particular rock structure. In case of the lack of fracturing, the direction of injected water flows in the formation is determined by the direction of predominant rock pore channels. It can be explained by different pore orientation in different directions. Thus, there is an anisotropy factor. The anisotropy of filtration properties in vertical and horizontal directions of sediment rocks is well known [1]. Such anisotropy is characteristic for all sediment rocks. In general, it is associated with sediment accumulation, secondary transformations during the process

of catagenetic changes and the impact of rock pressure. The anisotropy effect is clearly seen in petrophysical studies in different permeability factor values measured in parallel or normally to bedding. It is shown [18, 19] that in general, anisotropy has a tensor behaviour, appearing both in vertical and horizontal directions. Hence, the analysis of lateral anisotropy is of special interest. The anisotropy of filtration properties occurs also in carbonate rocks and, as a rule, is associated with fracturing [2]. However, when there is no fracturing in carbonate rocks, the effect of anisotropy of filtration properties is practically always present. It is particularly revealed in large-pore and cavernous interlayers. In plans of deposit development, vertical-horizontal filtration anisotropy is taken into account on the basis of geophysical data and petrophysical core studies. The azimuth anisotropy is poorly studied and due to the complexity of its evaluation it is not usually taken into account.

Theoretical analysis

Recently, due to the necessity of making more accurate 3D hydrodynamic field models, both vertical and azimuth (horizontal) anisotropy of filtration properties should be taken into consideration. The reasons for such anisotropy are the rock formation particles and pore space orientation as a result of rock formation and transformation, and the impact of tectonic processes. Detailed study of anisotropy of fluid conductivity requires petrophysical studies on oriented cores. The core orientation is available in a core sampling process during the drilling with special equipment

or on the basis of paleomagnetic methods which allow to orient the core in the space and to determine not only its current location in rocks, but its location during the rock formation [3]. At present there are works on the study of azimuth orientation of fluid migration in reservoirs, performed with oriented cores [4, 5, 6]. In this study [4], the authors performed a series of tests on anisotropy of the oil reservoirs using petrophysical, paleomagnetic and physical (elastic and magnetic) methods. It was shown that anisotropy of physical properties in rocks is caused

not only due to the changeability of petrophysical characteristic (permeability, secondary porosity), but also by the orientation of matrix decompaction (direction of change), its deformation due to fluid tectonic processes. In works by [7, 8] (Gurbatova I.P., Mikhailov N.N.), the anisotropy of properties of gas permeability and elastic wave velocity studies were performed on full-size carbonate rock samples. These experimental studies showed that besides vertical anisotropy there is also azimuth anisotropy in a bedding plain, typical for the studied samples collections, and the permeability in different directions differs strongly. The experiments showed that in some of the examined samples there was no direct correlation between oriented permeability and oriented velocity of elastic wave propagation. In general, the authors have found nonlinear correlation between azimuth-oriented permeability and propagation time of elastic waves. To explain the obtained substantial results, the authors suggested that there are two types of symmetry in the complexly constructed reservoirs: primary (sedimentary) and secondary, when the primary symmetry elements are overlaid by the processes of leaching and cavity formation. They define the difference in anisotropy of various physical properties. In the works [9, 10], a detailed

electron microscopy study was performed of clay rocks on the basis of Fourier analysis. The authors studied the microstructure orientation of clay rocks on the images obtained with a scanning electron microscope (SEM). The obtained data showed that the electron microscopy methods on the basis of digital image processing are highly informative for a study of microstructure orientation in clay rocks. In the work [11] (Kuzmin V.A. and Skibitskaya N.A.) were the first to perform such studies of carbonate rocks on the basis of the Fourier analysis of electron microscopic images. They studied the microstructure orientation influence on the pore space orientation and obtained the correlations between the degree of anisotropy in microstructure anisotropy and the degree of anisotropy of the of pore inter-granular space. It was found out experimentally that porous samples are nonhomogeneous in terms of azimuth orientation of microstructure and pore channels. It was found out that the degree of pore space orientation is related to a crystal matrix orientation in different ways. When porosity is comparable, more permeable samples have more oriented pore channels. The studies also showed that in some cases there are different orientations of microstructure particles and the pore space.

Procedure and experiment

To study lateral anisotropy, special experiments have been performed on full-size cores to examine orientation dependence on maximum permeability. In table 1 the lithologic characteristic of studied samples is listed. The studies were made with special equipment which allowed to determine permeability and time step in the normal direction to the vertical axis of the core. All measurements were carried out with a measurement step of 30° over the sector of the circle. Using the obtained data, the “leaf” diagrams (orientation roses) of permeability to gas and propagation time of elastic waves (ultrasonic time step) have been made.

In figures 1-3 the results of the studies in terms of the “leaf” diagrams (orientation roses) are presented.

As shown in the diagrams, all samples have different filtration and physical properties plus asynchronous change of permeability and time step values depending on the direction. In all samples there is also a difference between the orientation of the main filtration direction and the direction of time step propagation of elastic waves. The data for filtration anisotropy, carbonate rocks composition and porosity with cavities and their omission are listed in table 2.

The data in the table demonstrate that the samples with comparable porosity 6.7÷9.07% differ in the anisotropy of

Table 1. Lithologic characteristic of rocks

Sample	Lithologic characteristic
461-2-09	Limestone with organogenic detritus impurity, non-uniformly dolomitic, stylolitic, fractured, locally porous, non-uniformly oil saturated
461-6-09	Secondary dolomite with relict organogenic texture, calcareous, stylolitic, porous-cavernous, locally fractured, strongly oil saturated. Ornamental texture, stylolitic, locally fractured, porous-cavernous
461-33-09	Limestone with negligible clay impurity, stylolitic, fractured, partially porous-cavernous, non-uniformly BOS pigmented. Stylolitic texture, fractured, partially porous-cavernous, spotted due to extremely low content of clay material

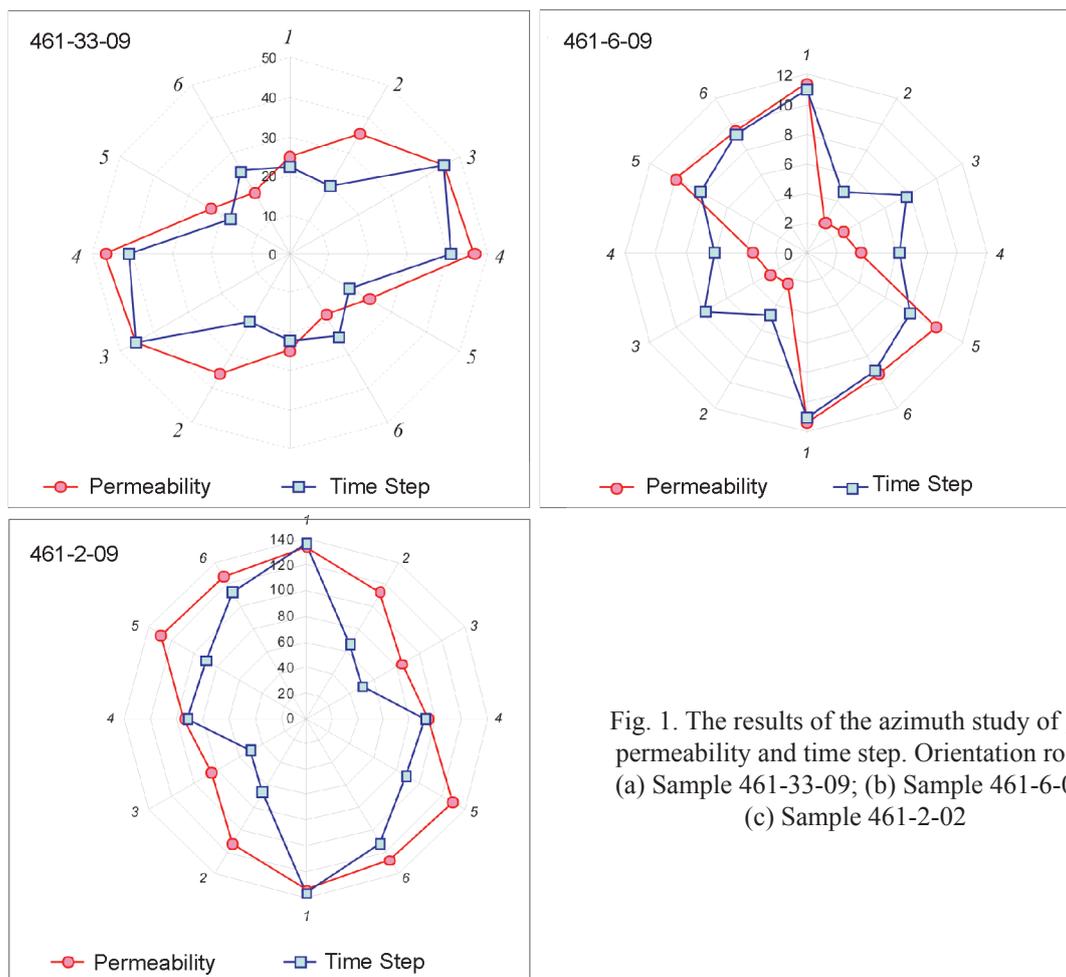


Fig. 1. The results of the azimuth study of gas permeability and time step. Orientation rose: (a) Sample 461-33-09; (b) Sample 461-6-09; (c) Sample 461-2-02

filtration properties (as well as permeability). In sample 461-6-09 the permeability value in azimuth direction (main and normal direction of filtration) is 5.44 and 5.24 mD respectively and there is practically no difference. The filtration anisotropy coefficient ($K_{a(f)}$) expressed as ratio of permeability difference to higher permeability would be 3.6%. The marked filtration properties anisotropy is seen in sample 461-2-09 with the gas permeability coefficient in the main and normal direction $K_{perm.}$ being 153.41 and 115.61 mD respectively and $K_{a(f)} = 32,7\%$. The maximum anisotropy of filtration properties is seen in sample

461-33-09, in which azimuth permeability differs practically twofold ($K_{a(f)} = 87.0\%$).

Since filtration occurs in the system of pore channels, in order to examine the reasons for such filtration differences it is necessary to study the features of rocks microstructure and their pore space at the micro level.

The auto emission scanning electron microscope (SEM) "LEO SUPRA 50VP" with field-enhanced cathode emission and 1 nm resolution was used for analysis in this work. Mineral composition identification was performed using X-ray spectral energy dispersion analyzer. Microstructure

Table 2. The characteristic of the anisotropy filtration-volumetric properties and rock composition

Laboratory sample №	Porosity			Gas permeability [mD]			Carbonate content [%]	
	Taking into account the external cavities	With ignored external cavities	External cavities fraction	Azimuth	Horizontal 90°	Vertical	Calcite	Dolomite
461-2-09	8.19	8.19	0	153.41	115.69	28.17	72.9	19.9
461-6-09	6.71	6.11	0.60	5.44	5.24	9.06	Not found	99.2
461-33-09	9.07	8.60	0.47	26.54	14.19	29.30	98.2	Not found

anisotropy was studied using the method of signal intensity gradient of sample chips SEM-images [12, 13]. Pore space analysis was performed by sections of the pores and channels on the surface of polished sections in SEM cathode luminescence regime using the Kuzmin procedure [14, 15].

Big pores and cavern scale in cavernous carbonate rocks are too large for studying by electron microscopy methods. The authors developed and used a method of contrast optical images for estimation of the degree of orientation (anisotropy in different directions). For this purpose, digital microphotography in contrast lighting of bedded samples surface was performed with a microscope MBC and a digital camera MDC20. For determination of integral values for crystal and pore orientation the above mentioned method of signal intensity gradient was used and realized with a program package. Essentially the method of the signal intensity gradients of SEM-images for determination of orientation is a modified method of analysis of the frequency of the change of image brightness gradation in different orientations, proposed earlier by Kuzmin V.A. (1978) [17]. The digital image processing algorithm is based on the estimation during the analysis of the local

signal intensity gradient V in every point of the image in two directions: dl/dx and dl/dy . The value for the gradient is calculated by



where l is a signal brightness in a given point of the image.

The signal intensity gradient is calculated by

$$\operatorname{tg} \theta \frac{dl/dy}{dl/dx}$$

The orientation rose is constructed using the analysis results. The length of its rays presents a relative value for the signal intensity gradient expressed in %. The direction of predominant orientation is defined in a normal direction of the maximum signal intensity gradient. The degree of orientation parameter is calculated by a resultant orientation rose and presents its anisotropy coefficient A , defined by

$$A = \left\{ 1 - \frac{(S_1 + S_2')}{(S_2 + S_2')} \right\} 100\%$$

where $(S_1 + S_2)$ and $(S_2 + S_2')$ are summed areas of radial segments along minimal and maximum axes of the orientation rose. The method is presented in detail in [10].

Results and discussion

The samples were studied in conventional SEM-regime (in secondary electrons) for the evaluation of the features of the rock microstructure. This regime allows the visualization of microstructure in a form of usual visual perception identical to optical means. During the study all the samples were oriented strictly along the main direction (direction of maximum filtration). The pore space was studied in SEM in a cathode luminous regime.

The SEM-data showed that all the studied samples are big-pore and big-pore-cavernous differences of rocks. Their significant volume fraction was formed by the secondary pores and caverns, mainly as a result of leaching processes. This means that in addition to smaller primary pores, there is a system of secondary pore channels of different genetic identity – intergranular pores, formed as a result of recrystallization, and large pore channels and caverns, formed as a result of dissolution of carbonate matrix. From the point of view of the orientation of the structural elements, differently oriented locations of the rock forming aggregates, crystals and grains are observed in all studied samples. A visual estimation of the trend of the crystal and pore orientation in SEM-images in most cases is impossible and consequently a visual estimation of microstructure anisotropy is impossible too. Thus, the

analysis of the “secondary-electron” images of microstructure showed that there are various microstructure morphologic, dimensional, micro textured and orientation differences of rock forming crystals and pore channels.

For comparison of intergranular pore space in the studied samples the histograms of pore size distribution (fig. 2) obtained by the method of cathode luminous SEM-images are presented below. For calculations [16] the program “Reservoir” developed in Oil and Gas Research Institute of the Russian Academy of Sciences was used. The given distributions cover only the range of the intergranular (inter crystal) capillary channels. In these distributions super capillary pore channels (cavernous channels) were not taken into account.

Judging by the behavior of the distribution, the histograms have a poly modal form with prevalence of big filtration pores. In all the samples an increase in a pore volume fraction is observed with the increase in their size. Such a distribution is typical of secondary porosity in carbonate cavernous rocks with high porosity. The largest intergranular pores are in sample 461-6-09 and their maximum size is 536,6 μm . It should be noted that the range of the size of secondary pores of the solution and intergranular pores, formed by crystal faces in coarse-grained, can be partially

overlaid, so the obtained histograms can be rather denoted as an intergranular pore volume fraction with a big degree of relativity.

In all the studied samples the filtration pore channels can be divided into two groups of different influence on the symmetry of physical properties (according to the Neumann principle). The results of electron-microscopic studies show that the orientation of intergranular capillary pores sized from 0.2 to 500 μm is related to the orientation of calcite crystals and dolomite in rocks. Larger super capillary pores formed by leaching have the orientation associated with other numerous factors affecting the degree and the rate of the solution.

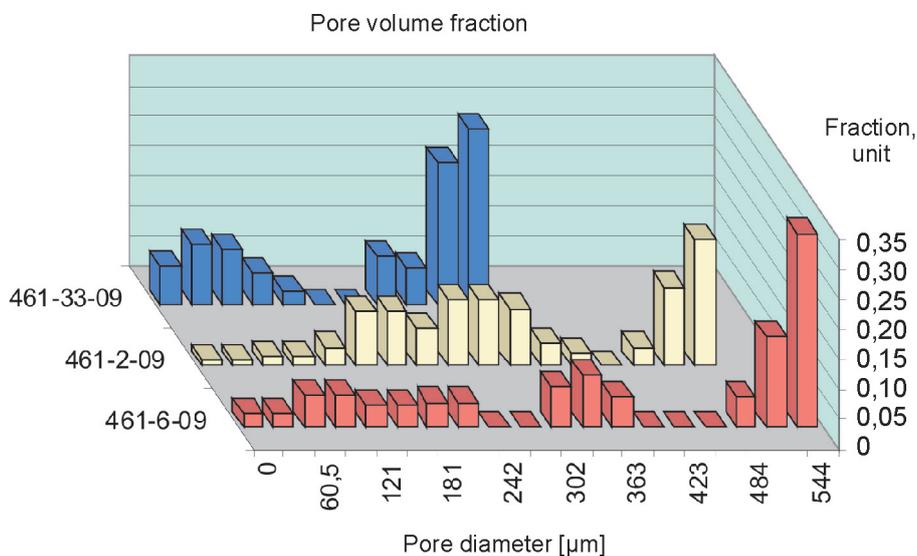


Fig. 2. Histogram of pore and channel volume fraction in the result of SEM-analysis

The study of azimuth orientation (anisotropy) of microstructure features in a carbonate rock pore space was performed in this work according to the following scheme:



a)

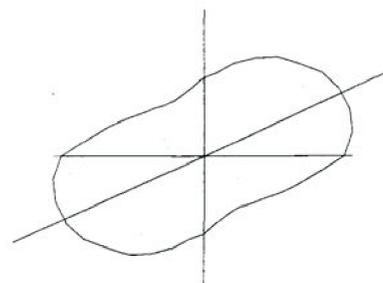
The large-pore space and caverns on the surface of the ground smooth plates cut from full-size cores were photographed in contrast lighting. Then, using appropriate software, the halftone images of pores and cavities were transformed into two-graded images. Digital images were used to obtain the anisotropy coefficient.

The microstructure of chips prepared parallel to bedding was studied in SEM in a "secondary electrons" regime. Next the analysis of the anisotropy and integral value for grain and carbonate matrix crystals was performed with informative enlargement using the SEM-images.

The polished sections impregnated with cathode luminophor were studied in SEM in a cathode luminous regime and the obtained images were analyzed to estimate the anisotropic properties and the pore space orientation.

Such an approach allowed to study in detail the influence of the microstructure factors on anisotropy and to reveal alternative pore channels directions by their orientation depending on their genetic identity and dimensional characteristics (intergranular and cavernous pores and channels). As an example, detailed study of the orientation and anisotropy of microstructure features and pore space of sample 491-2-09 is presented below. The estimation of

a large-pore space anisotropy and predominant orientation of pore channels was made with the digital analysis of optical contrast image of the ground smooth surface.



b)

Fig. 3. Sample 461-2-09. a) Photograph of the plate's section of a full-size core, the main filtration direction is at 25.5° with the horizontal (shown by the arrow) magnification = $\times 5$; b) orientation rose of large-pore channels. $K_a = 57.78\%$, angle of integral orientation is 25.0° . Deviation from the main direction of filtration is 0.5°

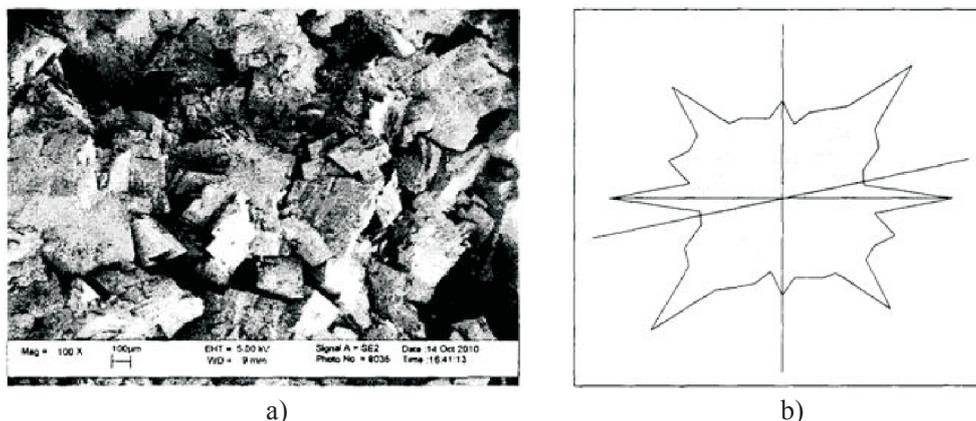


Fig. 4. Sample 461-2-09. a) SEM image of rock microstructure on which the analysis of orientation is done. Enlargement 100; b) orientation rose of microstructure elements (20-sector diagram). $K_a = 25.87\%$, angle of integral orientation = 73.72 degrees. Deviation from the main filtration direction is 48.22 degrees

A photograph of a large-pore space of the section of the plate made out of a full-size core where the azimuth estimations based on filtration and time-step is shown in fig. 3. The main filtration direction is shown by the arrow which forms 25.5° with the horizontal.

From the orientation rose it can be seen that in sample 461-2-09 there is practically a full coincidence of physical properties orientation (main direction) and (a) large-pore space orientation. The estimation of the anisotropy and orientation of microstructure was made by the secondary-electron SEM-images (fig. 4).

As seen from the orientation rose, for microstructure (crystals and aggregates) the angle of predominant orientation is at 73.72° with the horizontal; i.e. it has a 48.22° deviation from the predominant filtration direction (main direction). The anisotropy coefficient $K_a = 43.25\%$.

The analysis of capillary intergranular porosity was performed with cathode luminous images, which were partially defocused to obtain the halftones and improve the informative analysis. The values for K_a and integral orientation were calculated by the program method (fig. 5).

Identical studies were performed on samples 461-6-09 and 461-33-09. The generalization of data obtained from the studied samples shows that in case of high filtration anisotropy (samples 461-2-09 and 461-33-09) there is a significant anisotropy of cavities and large-pore channels with more than 500 µm in size. Intergranular channels have relatively low anisotropy (within 10%), but with significant angles of deviation of integral orientation from the main direction of filtration; i.e., there are alternative directions of the system of pore channels of different genesis.

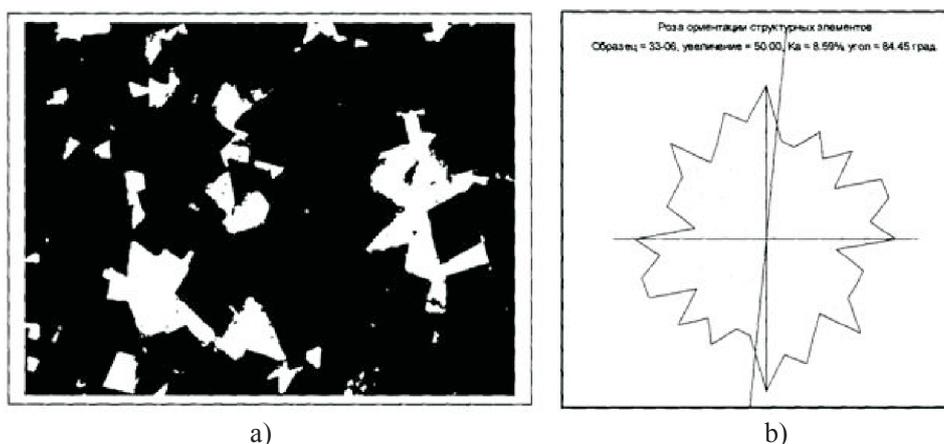


Fig. 5a. Sample 461-2-09. a) Image of a pore space of carbonate rock matrix. Enlargement = x50. Orientation rose (“leaf” diagram of 20 sectors), obtained by the analytical method of signal intensity gradient $K_a = 8.59$, the angle of integral orientation is 84.45°. Deviation from the main direction of filtration is 58.95°

For fig. 5b. Orientation rose of structural features. Sample = 33-06, enlargement = x50, $K_a = 8.59\%$, angle = 84.45°

In the sample with uniform filtration properties in azimuth direction (sample 461-6-09), the orientation of cavities and intergranular pore space is 6.19%, i.e. it is practically absent. For generalization of the obtained data

the results of the study of anisotropy of microstructure features and the pore space of the samples and measured angles of deviation of the direction of integral orientation from the main direction of filtration are listed in table 3.

Table 3. Results of the study of anisotropy of microstructure features and pore space of samples

Sample №	Optical method with contrast lighting		Scanning electron microscope (SEM)		Cathode luminous SEM		Anisotropy of filtration properties $K_{a(f)}$ [%]
	Orientation of large pores and cavities		Orientation of crystals and calcite and dolomite aggregates		Orientation of small pore (tentatively)		
	K_a [%]	Angle of deviation from main direction, grad	K_a [%]	Angle of deviation from main direction, grad	K_a [%]	Angle of deviation from main direction, grad	
461-2-09	57.78	Coincident	25.8	48.22	8.59	58.95	32.7
461-6-09	6.19	Coincident	23.0	Not orient.	3.14	Not orient.	3.6
461-33-09	44.41	11.92	59.9	5.23	9.72	13.6	87.0

Discussion of the results

The filtration anisotropy is closely related to the microstructural parameters of the rock. In the examined samples the main reason for anisotropy of the filtration characteristic is anisotropy of the pore space, where the leading role is played by the system of large-pore channels ($> 359\div 500 \mu\text{m}$), their orientation differs from the orientation of a smaller system of channels with matrix capillary intergranular porosity. There is no direct relationship between the coefficient of anisotropy of large-pore channels obtained by the method of image analysis and the coefficient of permeability anisotropy due to simultaneous influence of the whole pore system on filtration. The orientation of cavernous channels is poorly related to the orientation of crystals and carbonate matrix grains. The reservoir samples where there is no filtration anisotropy (sample 461-6-09, dolomite) have practically non oriented large-pore and intergranular pore space. In samples 461-2-09 and 461-33-09 the integral direction of a large-pore space orientation either coincided with the main direction of filtration, or was deviated by a small value of 11.92° in sample 461-33-09. In all the examined samples there is a microstructural anisotropy of a crystal rock matrix within $K_a = 23.0\div 59.9\%$ associated with integral orientation of the rock forming crystals and aggregates (grains). The angle of deviation from the direction of maximum filtration is $5.23\div 48.22$ degrees.

The performed studies showed that the application of scanning electron microscopy and digital analysis of images for problems associated with anisotropic properties of

oil and gas bearing rocks is highly informative. Performed studies revealed that the symmetry features of orientation in carbonate rocks are such microstructural features as pore channels, grains, crystals and aggregates of different composition present in the rock. It is known that according to the Neumann principle the symmetry of any physical property should involve the features of symmetry of the studied material. Therefore, there is a necessity to point out a pore space as a separate structure; i.e. the Neumann principle should be applied to the microstructure and the pore space separately. It is explained by the fact that the orientation of crystals and rock grains does not lead to synchronous orientation of the whole pore space. As it was shown by the electron microscopic studies in carbonate rocks of complex structure a large-pore and cavity pore space (of $> 0.5 \text{ mm}$ in size) may have an orientation that differs from the orientation of capillary intergranular pore channels (of $0.0002\div 0.5 \text{ mm}$ in size). It can be explained by the fact that cavernous porosity can be formed in a process of such secondary changes as dissolution under pressure and temperature. Dissolution processes for carbonates are extremely sensitive to structural non uniformities and other numerous factors affecting a rate of dissolution (non uniform distribution of minerals with different stability to dissolution, presence of impurities, solution compositions etc.). It leads to the formation of large-pore channels that were also formed by secondary processes, but at an earlier stage, as the result of diagenetic and catagenetic recrystallization. The orientation of intergranular capil-

lary pore channels depends on the nature of crystal and carbonate matrix grain packing. It should be noted that carbonate rocks of complex structure and high porosity as a rule have multi-level system of pore channels with different range of sizes. In cavernous rocks the system of intergranular pore channels may have a less fractional contribution in permeability than the system of large cavernous channels. As it has already been mentioned, at the same time an integral orientation of cavern channels formed by dissolution may have another direction that differs from the orientation of the system of intergranular pores. In this case, anisotropy of filtration properties and time-step will have alternative directions. According to the information obtained from the studies with SEM, the pore channels connectivity is an important constitutive part of fluid permeability anisotropy in different azimuth directions as well. It is explained by the fact that filtration is dependent on a pore connectivity. As determined in earlier studies, in the pore channels the orientation affects the pore connectivity in different directions and, as a rule, it is higher in a predominant direction of orientation. Seem-

ingly, a pore space connectivity changes as a result of rock compaction and decompaction under tectonic stress action. However, the problem of pore channels connectivity in different directions requires a more detailed study of the microstructure on oriented cores.

Thus, it can be concluded, that the direction of the maximum rate of filtration in the studied carbonate rocks is determined by the fractional permeability of intergranular capillary pores and large solution pores with different integral orientation. It was determined in performed studies that in carbonate rocks there is an orientation of rock forming calcite and dolomite crystals and the pore space orientation, which determine variability of filtration characteristic in different azimuth directions.

The results obtained in this work have a practical application in oil extraction, since if the azimuth orientation of rock pore space is not taken into consideration, it will lead to deviation of fluid flows during water injection from designed values. It decreases the sweep efficiency of the oil-producing formation and increases the volume of residual oil left in unaffected regions.

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Vladimir KUZMIN – PHD in Geology, author of over 130 scientific works and 10 inventions. Participation in the Moon rocks study in frame of the "Apollon" programme. Scientific interests: the phase transformation study of the rock Microstructure by the methods of electron microscopy.



Nikolay MIKHAYLOV – Professor of the Oil Reservoir Development and Exploitation Department and Gubkin Russian State University of oil and gas, Doctor Technical Sciences, Member of Russian Academy of Natural Sciences, Honorary Oilman of Russian Federation. The author is more than 120 scientific works. Scientific interests: i.e. theory and methods of oil and gas field development.



Vyacheslav MAKSIMOV – doctor of technical sciences, professor, Honoured worker of science of RF, deputy R&D director of the Oil and Gas Research Institute of RAS, the Gubkin Prize laureate, author of over 300 scientific works, including manuals, monographs and patents. Scientific interests: i.e. transfer process in porous media and theory and methods of oil and gas field development.



Irina GURBATOVA – Chief of the sample study department of Oil Company "Lukoil"; the author is more than 20 scientific works.