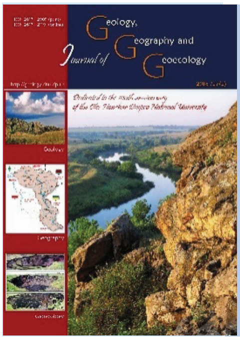

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The results of the research of sandstones closed porosity

V.A. Baranov, Y.V.Antipovich

Institute of Geotechnical Mechanics named by N. Poljakov of National Academy of Sciences of Ukraine, Simferopolska str., 2a, 49005, Dnipro, Ukraine, e-mail: office.igtm@nas.gov.ua

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Abstract. The article presents the results of the study of gas inclusions in quartz grains of Donets Basin sandstones. It describes the main genetic types of inclusions, their conditions of formation, shape, size, as well as features of the transformation under the conditions of catagenesis of rocks.The presence and distinctive features of the primary and secondary inclusions were established by studying of their homogenization temperatures. Thanks to these studies, it was found that the homogenization temperature of the primary inclusions is 1.5–2 higher than for secondary ones. Also, characteristic of the primary inclusions is that they are mainly gas-liquid. In size, primary inclusions are 1.5–2 times larger than secondary ones, and on average they are 2–3 µm. Secondary inclusionsessentially consist of gas. A characteristic feature of secondary inclusions in quartz grains of the sandstones of the Donetsk Basin is that they decorate the microdeformations of these grains, thereby forming numerous Boehm stripes. The gas inclusions of Boehm strips are indicators of paleotemperature, and the Boehm strips themselves carry information about paleopressure. To calculate the volume of gas inclusions, a methodwas proposed.This method is easy to use and does not require significant financial expenses. For its implementation, standard petrographic thin sections are used, which are examined using an optical microscope with a total magnification of 1000-1200 times. Using the proposed method, the volumes of gas inclusions in quartz grains of sandstones of different substages of catagenesis were established. Considering that gas inclusions are part of closed porosity, it is proposed to conditionally subdivide closed porosityinto cement closed porosity (volume of closed pores in the cementing substance of the rock) and grain closed porosity (volume of closed pores in detrital grains of the rock). It is established that the indicator of grain closed porosity is 2-3 times higher than the indicator of cement closed porosity. The largest volumes of gas inclusions are established for the middle substage of catagenesis. Considering that rock and gas outbursts occur only at the middle substage of catagenesis, this confirms the theory about the additional effect of gas inclusions on the progress of gas-dynamic phenomena in mine opening.Also, duringthe determination of absolute porosity of rocks in laboratory conditions, it is very important to grind the rock to micron-sized fraction. This will allow opening a part of inclusions in the clastic grains of rocks, which will substantially complement the indexes of absolute porosity.

Keywords: gas inclusions, microdeformations, catagenesis, closed porosity, sandstone

Результати дослідження закритої пористості пісковиків

В.А. Баранов, Я.В. Антіпович

Інститут геотехнічної механіки ім. М.С. Полякова Національної академії наук України, вул.. Сімферопольська, 2а, 49005, Дніпро, Україна, e-mail: office.igtm@nas.gov.ua

Анотація. В статті наведено результати дослідження газових включень в кварцових зернах пісковиків Донецького басейну. Описані основні генетичні типии включень, умови їх утворення, форми, розміри, а також особливості перетворення в умовах катагенез упорід. Присутність та відмінні особливості первинних та вторинних включень вдалося встановити завдяки дослідженню температур їх гомогенізації. Завдяки цим дослідженням встановлено, що температура гемогенізації первинних включень в 1,5-2 рази більша ніж у вторинних включень. Також, характерною ознакою первинних включень є те, що вони переважно газорідинні. За розмірами первинні включення перевищують вторинні в 1,5-2 рази, та в середньому досягають 2-3 мкм. Вторинні включення є переважно газовими. Характерною ознакою вторинних включень в кварцових зернах пісковиків Донецького басейну є те, що вони декорують мікродеформації цих зерен та створюють численні смужки Бьома. Газові включеннясмужок Бьомає індикаторамипалеотемператур, а самісмужкиБьоманесутьінформацію про палеотиск. Для визначення об'ємугазовихвключеньв кварцових зернах пісковиків, запропонована методика, яка є простою у використанні і не вимагаєзначнихфінансовихвитрат. Для її реалізації використовуються стандартні петрографічні шліфи, які досліджуються за допомогою оптичного мікроскопу з загальним збільшенням в 1000-1200 крат. За допомогоюзапропонованої методики встановленооб'ємигазовихвключень в кварцових зернах пісковиківрізнихпідстадій катагенезу. Оскільки газовівключення є частиною закритої пористості, запропоновано умовно виділятицементнузакритупористість (об'ємзакритих пор уцементі породи) і зернову (об'ємзакритих пор в уламкових зернах породи). Встановлено, щопоказникзерновоїзакритоїпористостіперевищуєпоказникцементноїзакритоїпористості в 2-3 рази. Найбільші об'єми газових включень встановлені для середньої підстадії катагенезу. З огляду на те, що викиди порід і газу відбуваються тільки на середній підстадії катагенезу, це підтверджує висунуту раніше вченими теорію про додатковий вплив газових включень на газодинамічні явища в гірничих виробках. До того ж, при визначенні загальної пористості порід в лабораторних умовах, дуже важливо подрібнювати породу до частинок мікронного розміру. Це дозволить розкрити частину включень в уламкових зернах порід, яка суттєво доповнить показники загальної пористості.

Ключові слова: газові включення, мікродеформації, катагенез, закрита пористість, пісковик

Introduction. Scientists have been studying gas inclusions in minerals of various genesis since the 18th century. The significant contribution to this field was made by the works of N. Ermakov (1972), E. Roedder (1987), V. Kalyuzhny (1982), G. Lemlein (1973), V. Baranov (1989) and others. These studies allowed establishing the location form of inclusions in minerals of different genesis, their origin, regularity transformation, consistence, as well as homogenization temperatures and morphological changes. Studies of inclusions were aimed at determination the pressure and temperature of minerals and rocks formation, detection of geological transformation stages, indications of ore occurrences, etc. (Tarantola, Diamond, Stünitz, Thust, Pec, 2012).

In the 70s-80s of the last century, scientists who were researching gas inclusions in minerals began to pay attention to gas-dynamic phenomena in sandstones, salt, and other rocks. According to E. Roedder, the cause of gas-dynamic phenomena in salt mines is the natural decrepitation (explosion, opening) of gas inclusions in thousands of tons of salt, which led to rock outburst in the mine workings (Roedder,1987).

During the last years, scientists have significantly increased the interest in carbon deposits of Donets Basin. According to the scientists' point of view, the Donetsk Basin should be considered as a coal gas deposit with complex exploitation to reduce gas emissions into mine workings and obtain unconventional type of gas materials at an industrial scale as well (Zhikalyak, 2002).

The main collectors of free gas in the Donets Basin are sandstones. They are associated with gas accumulations of industrial significance and the numerous micro accumulations of free gas. Despite this, the gas production is associated with a number of difficulties. The complex mining and specific geological conditions of basin deposits had a significant impact on deterioration of reservoir properties of sandstones. Significant resources of methane and poor reservoir properties of rocks lead to rock and gas outburst in the mine workings.

The study of the sandstones of the Donets Basin at the micro level has made it possible to reveal numerous gas inclusions in quartz grains of rocks. It was established that most of them are represented by methane, carbon dioxide and nitrogen (Baranov, 1989). According to V. Yakshin (1975), the volume of gas inclusions in gangue quartzreaches 10 cm^3 or more per 100 g of this mineral. The gas pressure in the inclusions reaches 500 MPa (Naumov, 1980). The huge amount of gas inclusions in the quartz grains of the sandstones of Donets Basin, and the possible pressure under which they are located, allows us to consider them as an additional factor affecting on theprogress of gas-dynamic phenomena in coal mines.

The main aim of this work was to study gas inclusions in clastic quartz of gas-bearing sandstones of the Donets Basin, their typification, to determine conditions of formation and transformation of this inclusions, establish their volumes, as well as the substantiation of practical significance of the obtained results in the context of studying the rock outburst and reservoir properties of sandstones.

Materials and methods. During the investigation, sandstone thin sections were studied. Samples of sandstone were taken within the distribution of different grades of coal. This made it possible to analyze sandstones of different stages of transformation. It is known that the transformation of sandstones of Donets Basin occurs under the influence of catagenesis and tectonic load. The substage of catagenesis is conditionally determined by the adjacent coal rank. Sandstone sections were examined using a POLAM R-111 optical microscope at magnifications from 100 to 1200 in reflected, transmitted, and side illumination. Data processing was carried out using personal computer.

Main part. Gas inclusions in the quartz grains of Donets Basin sandstones are represented by two genetic types - primary and secondary inclusions.

Primary inclusions were formed at the time of crystal formation in the parent rocks. They are well identified by optical research, since they are mainly two-phase and consist of liquid and gas phases (Fig. 1). In general, in the quartz grains of the sandstones of the Donets Basin, their amount is 1-2% of the total amount of all inclusions. The characteristic feature of primary inclusions is their shape. With an increasing of the stage transformation of rocks and tectonic load, the primary inclusions acquire the structural or morphological form of the host mineral. The size of primary inclusions usually exceeds the size of secondary inclusions, on average 1,5-2 times. The size of the primary inclusions in the quartz grains of the sandstones of Donets Basin on average 2-3 μm.

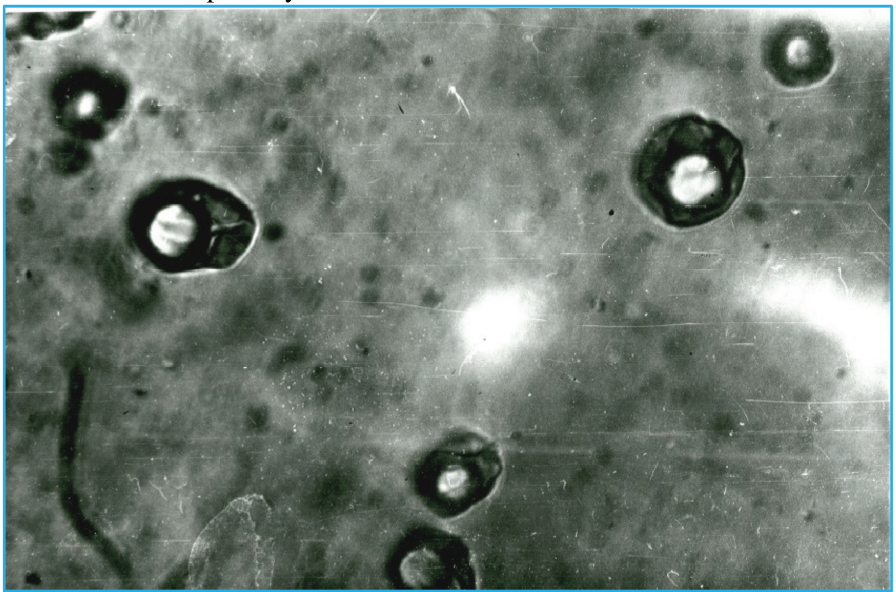


Fig.1. Primary gas-liquid inclusions in quartz of Donets Basin sandstones, 250^X magnification

Secondary inclusions are formed in early catagenesis, initially in the form of plastic and brittle deformations into which fluid from intergranular pores penetrates. Further, defects filled with fluid are healed, forming fluid inclusion chains. They were first described by Augustus Boehm in 1883, in the Alpine deposits. Later, these defects were called “Boehm stripes”.

Thus, the secondary inclusions in the quartz grains of the sandstones of Donets Basin are presented in the form of Boehm stripes (Fig. 2). Boehm stripes are presented both in catagenesis, in terrigenous sediments, and in metamorphic and igneous rocks. But with the transformation of rocks from one stage to another, for example, from sedimentary rocks to metamorphic or igneous, the inclusions are completely transformed or disappear, since the above processes are characterized by a complete structural reorganization of the rocks. In the study of sandstone sections, Boehm stripes are well identified by oblique illumination method (Baranov, 2018).

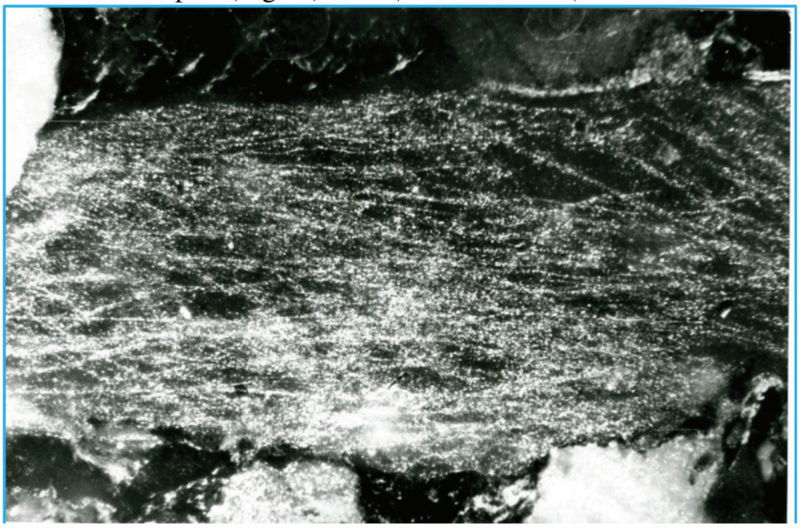


Fig. 2. Quartz grain with Boehm stripes, carboniferous sandstone of Donets Basin, oblique illumination method, 100^X magnification

Boehm stripes in the quartz grains of Donbas sandstones formed during the catagenetic transformation of sandstones directly in the conditions of the basin (Baranov, 1989). The prevailing development of Boehm strips in sandstone quartz grains is explained by increased gas content formed during the process of coal carbonization and dispersed organic matter in sedimentary rocks.

Sandstone, as the main reservoir, accumulates gas in open pores and cavities. During the transition from the early substage of catagenesis to the middle one, gas from the intergranular space migrates to microdeformations in the clastic grains of sandstones, thereby forming numerous chains of gas inclusions. Later on, at the middle substage of catagenesis, these microdeformations are healed and gas inclusions are altered. They acquire an isometric form, clearly identified in the optical study. The largest number of Boehm strips is found in quartz grains of sandstones from the middle substage of catagenesis. In the late substage of catagenesis, the size of gas inclusions and their number decrease. This is explained by the fact that, with an increasingofcatagenetic transformation and tectonic loading, the inclusions are divided to the smallest size and form pores of the size of hundredths and thousandths of microns. Subsequently, with an increasingof pressure and temperature, the gas from the inclusions migrates into zones with lower pressure, for example, into fractured zones or into intergranular space. Figure 3 showsthe changing of average size of Boehm strips inclusions under thecatagenesis effect.

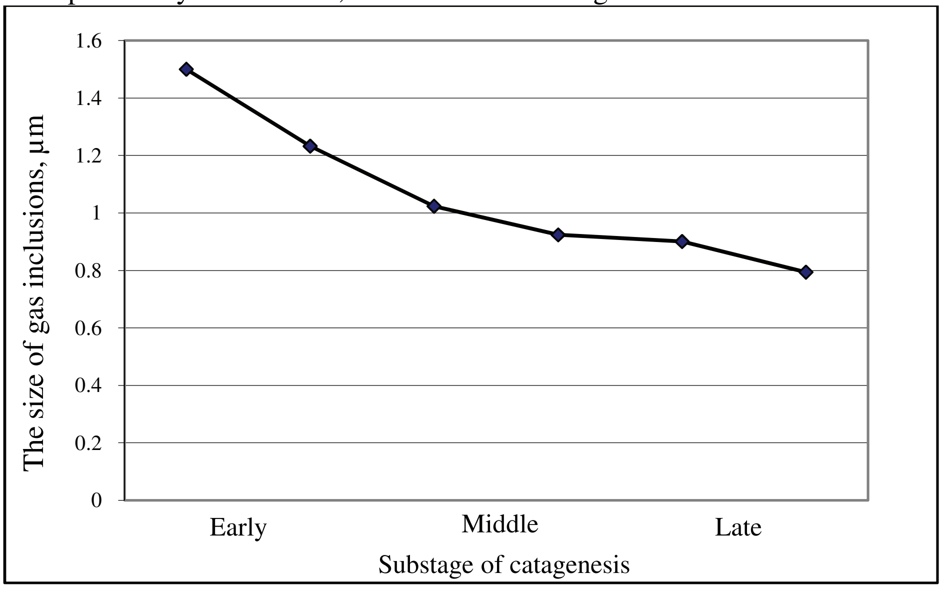


Fig. 3. Influence of the catagenesis on changing of gas inclusions size in quartz grains of sandstones

Boehm stripes are used as an indicator of paleopressures and paleotemperatures which will be described below.

Paleotemperatures. Currently, researchers obtain data concerning paleotemperatures by two basic methods – according to a degree of reflecting capacity of vitrinite and according to homogenization (decrepitation) of gas-liquid inclusions.

The presence and distinctive features of primary and secondary inclusions were established by studying the temperature of homogenization of inclusions in the quartz grains of carbonic sandstones of Donets Basin. Thanks to these studies, it was possible to establish that temperature of primary inclusions homogenization is considerably (by 1.5-3 times) higher than that of secondary ones. The latter belong to such microdeformation type as Boehm strips decorated with either gas or gas-liquid inclusions. Catagenesis temperatures obtained just with the help of secondary inclusions are quite homogeneous within similar conditions and, in terms of one and the same coal grades, vary within 5÷10^{0}C.

It is very important fact that we can see direct temperatures of the inclusions structuring. Temperature step or temperature gradient of each coal grade is 20^{0}C, ± 5÷10^{0}C. Sandstones were not analyzed thoroughly within a region of anthracite occurrence. Table presents the approximate temperature range at which the structures of the coal and the adjacent sandstones can be transformed.

Table. Paleotemperatures of the formation of different coal grades

Coal grades	High volatile	Medium volatile	Coking steam	Dry-steam	Antracite
Paleotemperatures, ^{0}C	80-100	120-140	160	180	200-300

The following may be used as an example. Sandstones (occurred in the neighborhood of coalbed) were sampled to measure paleotemperatures; their specimens were analyzed according to specific methodology. Temperature of samples taken near the high volatile coals grade was 100^{0}C. Temperature of sandstone sample taken in one of mines was 120^{0}C. After more than 10 years of the mine operation it has become known that its coal seam belongs to Medium volatile grade. Such a thing may happen occasionally due to the nonhomogeneous conditions of carbon matter formation; initial material; or various interpretation errors. The method to measure paleotemperatures can be much more accurate than thermometry in terms of reflecting capacity.

Paleopressures. Currently sensors and instrumental facilities are available to measure pressure in rocks, wells or mine workings are available (Liubimov, Nosenko, 1978). However, there is the problem of measuring paleopressures within sedimentary rocks to determine reservoir properties, to carry out stage analysis, and to solve specific problem – whether it is expedient to drill deeper while exploring carbohydrates or further drilling process has no prospects. It is knownthat along with the increasing depth and lithostatic pressure, porosity and permeability of rocks decrease as a natural result but sedimentary basins are characterized by vertical upward/downward movements. Hence, intensely consolidated rock with poor reservoir properties may go up close the surface and upper rocks will be washed down. How is it possible to determine a degree of paleopressures and possible availability of the reservoirs in such rocks? Below, we will consider methods to measure paleopressures within sedimentary rocks with the use of natural indicators of stressed state of rocks.

The method is based on the phenomenon of light flux deflection and reflection within the areas where matter continuity is disturbed. Direct light flux passes through homogeneous substance free from defects with similar density and velocity. While passing through the substance with volumetric defect, light flux is deflected within the point of defect location. Thus, separation of light flux into direct (in terms of defectless point) and deflected (in terms of defect point) takes place; the latter is behind the direct flux by certain length of light wave $\Delta\lambda$. That is the explanation of the available visible defects in the context of direct lighting in the form of dark points and bands. In the context of side lighting, only insignificant share of light beams falls onto the substance (e.g. quartz). Due to multiple deflections of light beams on their sides, volumetric defects become peculiar light concentrators demonstrating themselves in the form of light points and bands on the background of less lightened defectless field of the substance. Oblique illumination methodmakes it possible to demonstrate various gas-liquid inclusions, Boehm strips, bands and plates of deformation as well as other structural irregularities in more contrasting and clear form.

Using microscope, microdeformations in thin rock section are calculated and plasticity coefficient (Cpl) is determined as follows:

$$Cpl = (\Sigma N) \div (\Sigma n \times \bar{d}),\ \ \mathrm{mm}^{-1}, \qquad (1)$$

where *Cpl* is plasticity coefficient;

ΣN is total number of deformation marks calculated;

Σn is total number of the quartz grains studied;

$\bar{d}$ is average size of fragmental grains in the rock, mm.

Minimum quantity of quartz grains being calculated was determined empirically using the methodology developed by the S. Saltykov(1970). According to the methodology, total number of slip bands within each considered quartz grain was calculated; the values were columned. In parallel, values of accumulated average number of slip bands varied depending upon the number of considered grains were columned as well. Along with the increase in the number of the latter, variations of the accumulated average value becomes more and more narrow; it experiences its stabilization approximating the true value (Fig.4).

According to the results of experimental data, it is required to calculate 100-150 quartz grains to obtain deviation value in terms of 5-8 % average one. Such accuracy is quite sufficient to get error value within 10 % being adequate for the calculations. The developed index may be called a specific coefficient of rock-forming grains disturbance.

The index is not free from its disadvantages connected with the specific nature of petrographic definitions. One fragmentary grain may involve dozens of deformations with different lengths, widths, and contrast ratios; that is why a degree of accuracy depends upon the qualification of an operator. Sophisticated activities need a high-skilled operator. Attention should be paid to a specific nature of interpretations as well as all the possible nuances available in various devices and measuring techniques.

To improve both reliability and adequacy of the obtained results as well as rapidity, coefficient of relative disturbance Cd, % was developed later. The coefficient is determined by the formula (2).

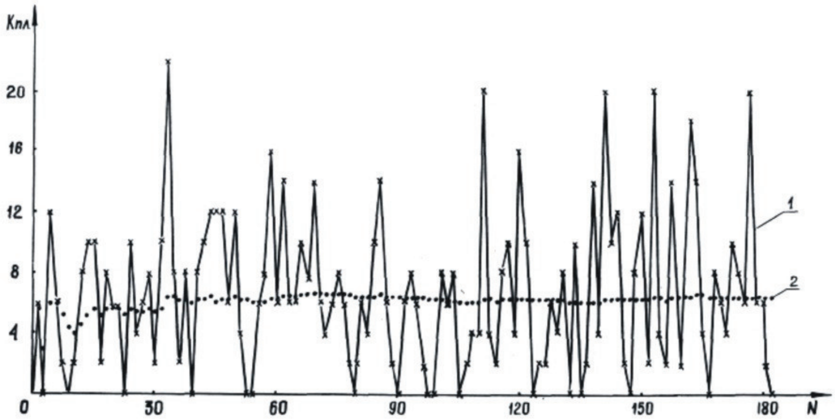
$$Cd = (\sum m \div \sum M) \times 100\ \%, \qquad (2)$$

where Cd is coefficient of relative disturbance;

$\sum$m is the total number of rock-forming minerals with the traces of plastic deformations;

$\sum$M is the total number of rock-forming minerals of sandy fraction analyzed.

The index is required to determined relative number of disturbed grains within the rock under study. As the previous one, it registers higher stresses in rock in the form of microdisturbances. The coefficient shows the number of disturbed grains within the rock under study demonstrating objectively those paleostresses experienced by the rock in the context of maximum subsidence or maximum tectonic movements. It can be applied while analyzing regional regularities of lithogenesis on the whole and catagenesis in particular, especially in the context of middle substage of catagenesis as the basic phase of carbohydrates formation



1 is the total number of slip bands within a grain; 2 is the values of accumulate average

Fig.4. Changes in the accumulated average number of slip bands*($K_{пл}$)*depending upon the total number of quartz grains calculated*(N)*

Moreover, the index has become highly applicable to identify disturbing zones as its values increase while approaching a disturbed zone. The idea is logical and it can be described easily in terms of following example. Imagine clear hard substance experienced an impact by a heavy thing; force of the impact can be determined relatively by the number of microfissures which can be identified in this case with the help of microscope. Since the disturbed zones within rocks can be direct intake channels for carbohydrates, we can extract the carbohydrates (if they are available there) by mounting perforated pipes.

Together with the porosity, the developed index was used to determine the degree and substages of rock catagenesis. That is of high importance both for miners (since outbursts of sandstones are connected only with the middle substage of catagenesis) and oilers as the reservoir properties of rocks depend heavily on their paleodepth occurrence. Modern depth coincides rarely with paleodepth; thus, if we know the history of reservoir formation, we may reason about their properties. Depth of 5 km cannot be the limit of reservoir availability taking into consideration the fact that in Dnieper-Donetsk aulacogen (Ukraine) at the depth of 3 km, driller have found loose deposits (Ignatchenko, Zaitseva, Ivanova, 1979), i.e. such depths are quite available for diagenesis deposits.

Investigation of gas and gas-liquid inclusions in clastic grains of rocks also has one more important practical interest. A significant pressure in the inclusions and their quantity create an additional volume that must be taken into account at the time of studying reservoir properties of rocks, in particular porosity. Determining the volume of gas inclusions in the clastic grains of rocks will help to establish an additional amount of closed microporesgas of the Donets Basingas-bearing sandstones.

In general, closed porosity of rocks is determined as the difference between absolute and open porosity. It is believed that closed porosity has only a scientific interest, so in practice it is ignored.

The disadvantage of determination the closed porosity by the calculation method, as the difference between absolute and open porosity, is that we only get the values of the volume of pores that is contained in the cement of the rock. The amount of gas that is represented as inclusions in the clastic grains of rocks is not actually taken into account, since in determining the total porosity by the grinding method, the rock is not always crush to the size necessary for the opening of these inclusions.

Considering that the inclusions in the clastic grains of rocks are also part of the closed porosity, it was proposed to divide the closed porosity into the cement closed porosity (the volume of closed porosity contained in the cement rock material) and grain closed porosity (the volume of gas inclusions in clastic rock grains).

To establish the volume of closed grain porosity, a method was proposed. The method is easy to use, economically advantageous, because it does not require additional expenses. To implement it, standard petrographic thin rock sections are used, which are made in laboratories of geological organizations to determine various indicators in minerals and rocks. The thin sections are examined on a POLAM R-111 type microscope. The study is conducted at a magnification of 1000-1200 times, using an object micrometer.

The essence of the method is that the volume of gas inclusion is determined by the ratio of the area of this inclusion to the area of the investigated part of in the clastic grain of the rock. Therefore, choosing an informative part of clastic grain in thin sandstone section, it is necessary to tie it to the correct geometric shape. In most cases, it can be square or rectangular. Using the well-known formulas for determining the area of a square or rectangle, it is necessary to determine the area of the investigated part of thin section. When the area of the investigated part is known, it is necessary to calculate the area of all inclusions that are in this part of clastic grain. The volume of gas inclusion with a diameter of less than 0,4 mm without a significant error can be taken equal to the volume of the sphere (Kalyuzhny, 1960). But since the sphere is a three-dimensional figure and a thin section is a two-dimensional subspace, we take each inclusion as a circle and calculate the area of inclusion through the radius of the circle. In the end, we calculate the total area of all inclusions in the part of the studied grain. In percent by the formula (3), we determine the volume of inclusions in the clastic grain of the rock.

$$V_{incl.} = \left(S_{incl.} \div S_{inv.p.}\right) \times 100\ \%, \qquad (3)$$

where $V_{incl.}$ istotal volume of gas inclusions;
$S_{incl.}$ is total area of inclusions;
$S_{inv.p.}$ is the area of the investigated part of thin section.

Using this method, it was found that the largest volumes of gas inclusions are characteristic of the middle substage of catagenesis and on average reach 4% (Fig. 5). The early substage of catagenesis is characterized by insignificant volumes, which constitute no more than 2%. This is due to the fact that the microdeformations in quartz and their gradual filling with gas take place in the early substage of catagenesis. The middle substage is characterized by the largest number of microdeformations. Gas inclusions that fill microdeformations acquire clear shapes and sizes. Sandstones of the middle substage of catagenesis are the most informative in studying inclusions in clastic grains. At the late substage of catagenesis, the volumes of gas inclusions decrease and reach 2%. This is due to the fact that in the late substage of catagenesis, under the action of high pressure, the structure of quartz begins to change. Monolithic grain turns into an aggregate, parts of which are cleaned, deformations and microinclusions go to the formed boundaries.

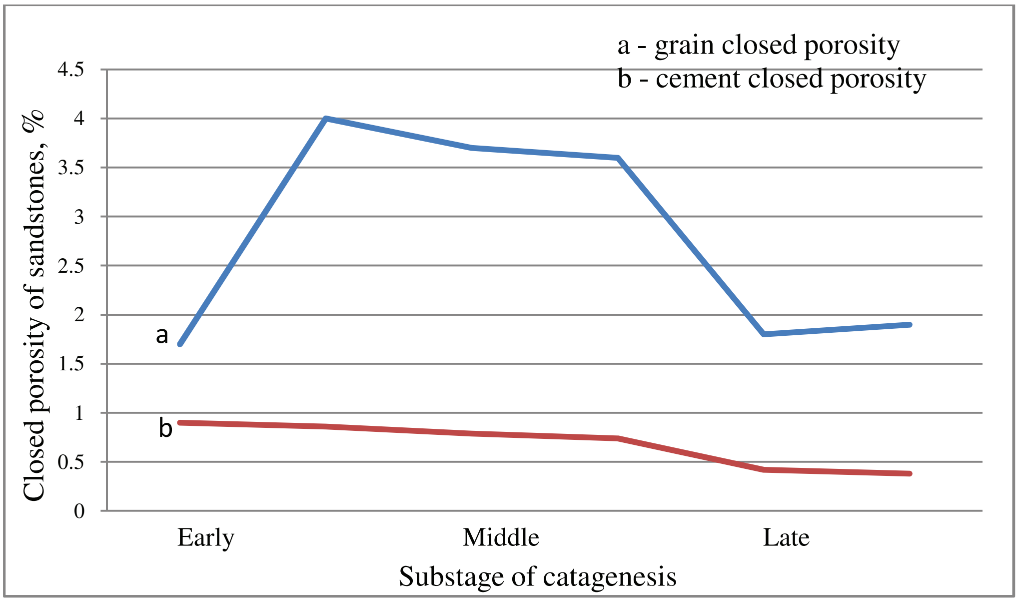


Fig. 5. Influence of the catagenesis on changing of sandstones closed porosity

It is important to note that the volumes of gas that we obtain using this method are not absolute. Conducting a study of thin sections with a magnification of 1000-1200 times the minimum size of inclusions, which we can observe, is 0.3 microns. Conducting research at larger magnifications would allow us to investigate smaller inclusions and, accordingly, establish additional gas volumes. This method is not aimed at establishing absolute values, but at establishing regularities of changing the volume of inclusions under the conditions in which the studied rock is located.

From the graph in fig. 5, it can be seen that the values of closed grain porosity are several times higher than those of closed cement porosity, which averages 1%.

The largest volumes of gas inclusions are established for the middle substage of catagenesis and reach 4%. Considering that rock and gas outburst occur only at the middle substage of catagenesis, this confirms the theory put forward earlier about the additional effect of gas inclusions on the gas-dynamic phenomena in mining workings.

The volume of gas inclusions must also be taken into account during the determination of the reservoir properties of rocks. Determining the absolute porosity in laboratory conditions, it is necessary to grind the rock to the size that would allow revealing the smallest inclusions in the rock-forming grains. Part of the gas contained in the rock-forming grains will substantially supplement the data of absolute porosity of the rocks.

For example, for sandstones of the middle substage of catagenesis (Stakhanov mine, Krasnoarmeysky district, Donets Basin), an average of 3.5% of gas in closed micropores of quartz grains was established. The absolute porosity of the studied sandstone is 7%, but considering the volume of gas in quartz grains, it can reach 10 - 10.5%.

Conclusion. The study of gas and gas-liquid inclusions in the clastic grains of rocks has an important scientific and practical interest. These inclusions involve genetic information about minerals, as well as being a source of data on secondary transformations of rocks. Examining the inclusions of the Boehm strips, we can obtain data about the paleotemperatures of rocks. Boehm strips are also a source of information about the paleopressure. The results of the study of gas inclusions in the quartz grains of the sandstones of Donets Basin allowed establishing the basic regularities of their transformation at different substages of the catagenesis. Considering that gas inclusions in clastic grains of rocks are part of closed porosity, it was proposed to subdivide closed porosity in cement closed porosity and grain closed porosity.It was determined that the closed grain porosity of sandstones is formed in the early and middle substages of catagenesis and in the late substage it is transformed. Cement closed porosity depends in large part on the mineral composition of the micas, the mineralogical form of carbonates, and in catagenesis it is transformed simultaneously with the change in the structure and form of these minerals. The developed method of calculating the volume of gas inclusions, allowed establishing that the volume of grain closed porosity on average is 1,5-2 times higher than the volume of cement closed porosity. These volumes must be considered as additional during the determination the reservoir properties of rocks and forecasting gas-dynamic phenomena in mining workings.

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