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## **ANALYSIS OF COAL STRUCTURE IN THE ASPECT OF GAS CONTENT**

### **1. INTRODUCTION**

Porous materials are characterized by the presence of solid material empty spaces (pores) of different sizes and shapes that are connected together and forming irregular network.

Porous structure can be a natural property of the material, as in the case of coal or is formed by chemical or thermal processing as is in the case of activated carbon. Commonly pores that are connected with the exterior surface are defined as open pores and empty spaces that do not have such a connection are defined as closed pores [1].

Coal is a porous material with pore sizes that span wide length scales including macro-, meso- and microporous regimes. The porosity plays a key role in all aspects of coal utilization, such as extraction of methane from coal seams, gasification, combustions, liquefaction, production of metallurgical coke and activated carbon as well as geological sequestration of CO<sub>2</sub>. The debate about the nature and structure of the pores in coal is ongoing. According to a widely accepted consensus, coal is a solid that contains slit-like pores interconnected by narrow capillary constrictions and connected to the surface. However, recent studies have suggested that a significant proportion of pores in coal may not be open to the external surface. It is not known if pores in coal are inaccessible to green house gases such as methane and carbon dioxide, and the issue of selectivity of access to pores of different sizes is even more obscure. However, such information is particularly important for the practice of ECBM (enhanced coal bed methane recovery), a technique that uses injected CO<sub>2</sub> to increase the extraction efficiency of methane from coal seams. Experimental data on pore accessibility and adsorption selectivity could help to understand the fundamental limits

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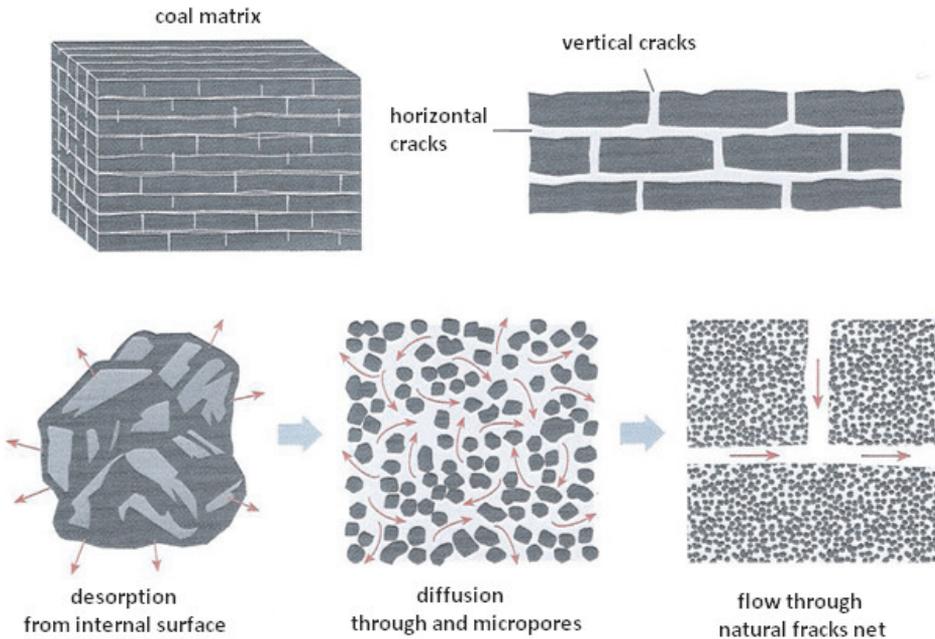
to the ability of  $\text{CO}_2$  to displace methane in subsurface conditions during sequestration of  $\text{CO}_2$  in coal seams [2].

The problem of methane existence in coal beds has been known for many years. It was and still is a danger to coalminers.

The presence of methane in coal is linked to the process of coal beds creation. It is believed that the methane in coal with a low degree of coalification is formed as a result of microbial activity, while it coals with a higher degree of coalification by thermal processes of organic matter. Organic matter was accumulated in swamps as a residue of plants growing on the Earth during periods when the climate was hot and the organic matter was subject to compression. As a result, a chemical and physical reactions, leading to the formation of coal, methane, carbon dioxide, nitrogen and water. As the layers of organic matter formed, pressure and temperature rise, leading to an increasing degree of coalification of the material and an increase in methane content [3].

Methane contained in the coal occurs [4, 5]:

- in the adsorbed form in micropores of diameter less than 2 nm,
- is detained in a matrix of carbon, bound with a weak van der Waals bonds to the coal surface,
- in the form of free gas trapped in fracks and cracks,
- as dissolved in the water contained in the coal.



**Fig. 1.** Process of methane recovery from coal [3]

In the process of release and flow of methane in coal the following phases can be marked [3, 5, 6]:

- phase lasted about two years, when the pressure is reduced in the rock matrix as a result of relaxation of the rock mass due to dehydration deck,
- methane desorption phase,
- phase of diffusion of free methane from the matrix into the cracks,
- flow in the natural frack phase.

Figure 1 shows a diagram of the construction of a coal bed in the decks and desorption and diffusion processes and process flow in the slots.

The gas contained in the coal is initially adsorbed onto the surface of the coal, as a result of pressure reduction, and more begins to form a continuous phase, followed by a further period of gas flow through the slots and cracks [3].

## 2. THE COAL DEMETHANION METHOD WITH THE USE CARBON DIOXIDE

The presented study of the coal microstructure is a part of the coal demethanation method with the use of liquid CO<sub>2</sub>, that was proposed by Military University of Technology as patent no P.402397.

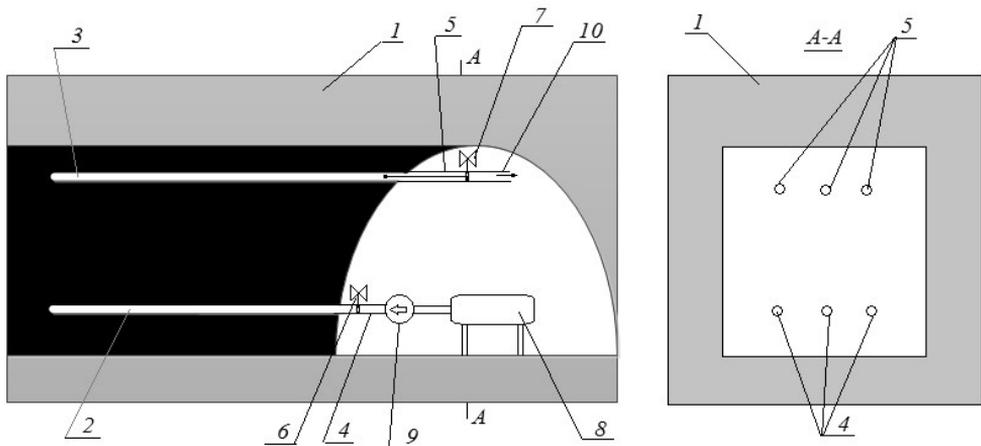


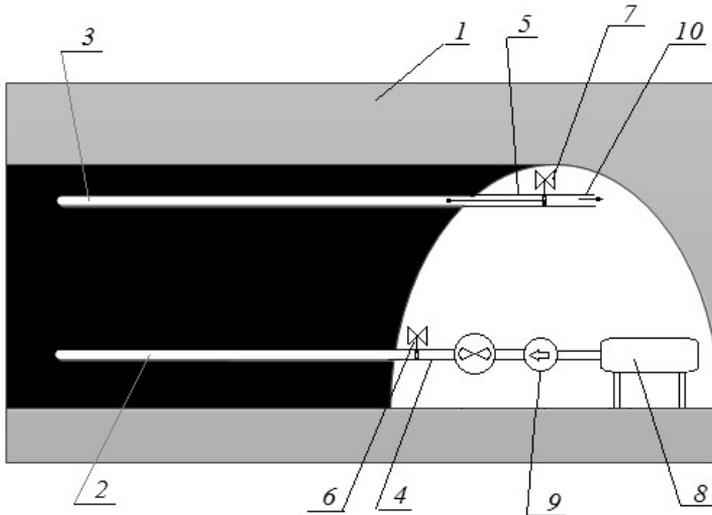
Fig. 2. Scheme of the proposed demethanation method

Scheme of the proposed method is shown in Figure 2. In intact coal bed 1 directional of small diameter (drainage) holes are drilled on at least two levels, the lower 2 and upper 3 ones. The pipes 4, 5 – suitably prepared (isolated, pre-cooled with liquid nitrogen or cooling jacket) are introduced into the holes. A cemented hole that is pre-cooled by liquid nitrogen can also be used. The shutoff valves 6, 7 are installed at the opening of the pipes. The valves 7 at the upper holes are equipped with a pressure sensor. The tank 8 for liquid CO<sub>2</sub> is placed

on the pipe of the lower level. The pump 10 is situated at the outlet of the CO<sub>2</sub> tank. Outlet pipes of the upper level are connected to the demethanation system of the mine 9.

The demethanation process is carried out in stages. At the beginning the valves 6 and 7 are closed. Precooled liquid CO<sub>2</sub> is located in the tank 8. After opening the bottom valve 6 carbon dioxide is pressed through the pump 10 to the lower level of the holes 2, and the valve 6 is closed.

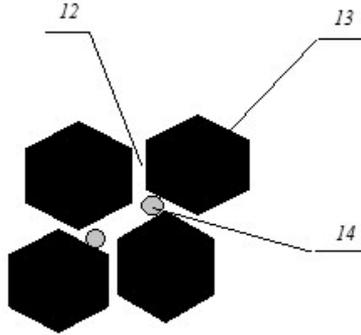
Carbon dioxide under the influence of the temperature in the coal bed extends and causes rock fracturing, which improves its permeability. In addition, carbon dioxide is adsorbed on the coal surface, causing a simultaneous desorption of methane, which additionally increases the intensity of the demethanation process. The process can be enhanced by using a mixture of carbon dioxide and sand, which will keep the cracks open. The use of sand has to be assessed in accordance to mechanical properties of coal. The scheme of the demethanation process with the sand – CO<sub>2</sub> blender 11 is shown in Figure 3.



**Fig. 3.** Scheme of the proposed demethanation method with carbon dioxide and sand blender

The use of sand to the carbon dioxide mixture causes the blocking of the cracks before they are closed. This process is shown in Figure 4, where the crack 12 between grains of coal 13 are carried by particles of sand 14. Efficiency of the process depends on the strength properties of coal.

Processes occurring in the coal bed are continuously monitored by pressure sensors in the valve 7 located at the upper level of holes 3 in order to prevent discharge of CO<sub>2</sub> during the fracturing process. After the time required for cracking of coal and desorption of CH<sub>4</sub> upper valve 7 is open and methane lighter than carbon dioxide is pushed to the upper level of 3 holes. Recovery of gas from the hole may occur spontaneously or be vacuumed. The recovered methane is released to the existing demethanation system in a mine 9 and uncaptured rest of methane flows into the air vent.



**Fig. 4.** Scheme of cracks blocking with sand

### 3. THE GOAL AND METHODOLOGY OF RESEARCH

The aim of the research presented in the paper is to show and assess the porosity structure (especially micro and nanoporosity) in accordance to the dimensions of carbon dioxide particle. The porosity and the size of micro and nanopores can decide about the amount of methane particles adsorbed on the coal structure surface. So the study on the proposed subject can give the key information to develop the most effective method of coal degasification.

Porous coal structure is associated with the presence of particular maceral. Results of chemical and maceral composition analysis of coal used for tests are presented in Table 1.

**Table 1**

Characteristic of the coal sample used for studies

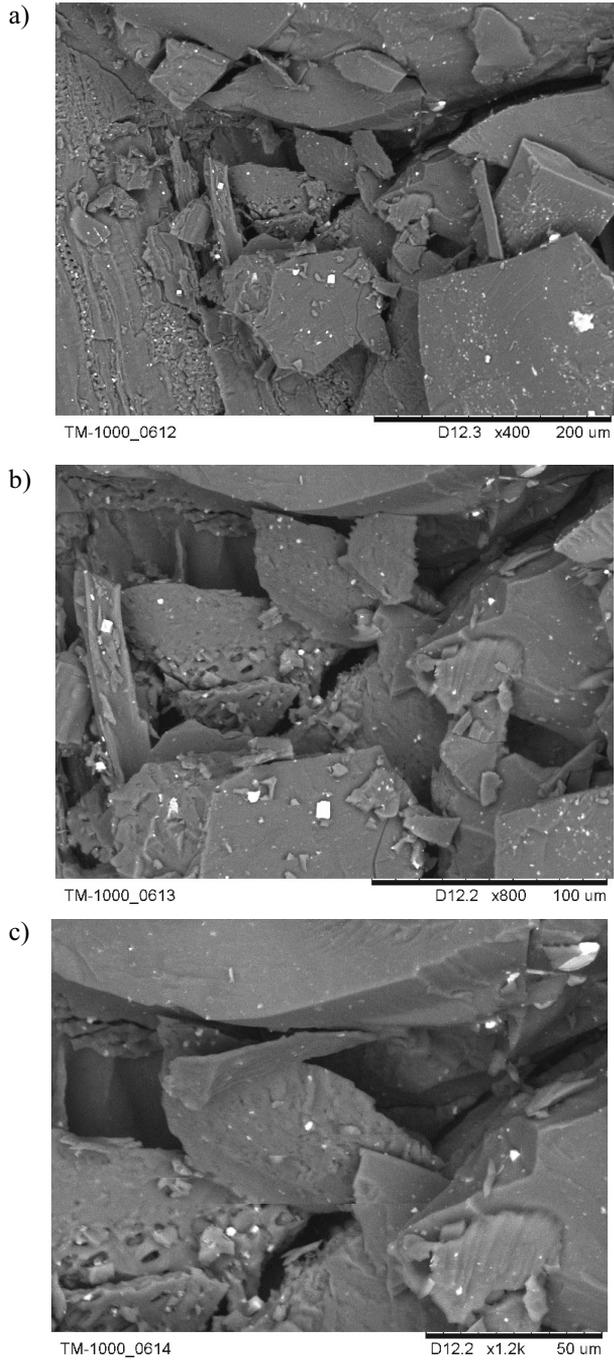
Coal	C <sup>daf</sup> [%]	Vitrinite reflectance, Rr [%]	Vitrinite [%]	Liptinite [%]	Inertinite [%]
A	85.6	1.14	72	6	20

Observations of samples for surface morphology in the initial state were carried out with the use of the Hitachi scanning electron microscope model TM-1000, using a magnification of 20 to 10 000 times. Accelerating voltage of 15 kV was applied. Samples were observed in contrast backscattered electrons (BSE) low vacuum mode (5 Pa).

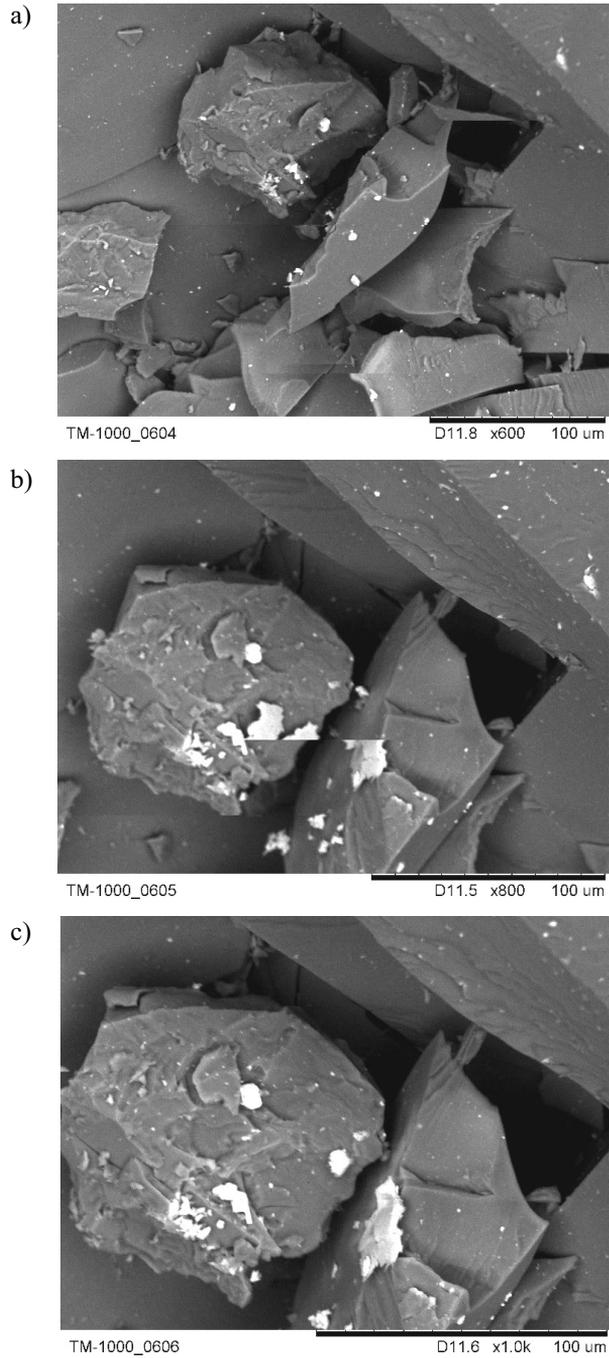
Observations of the samples for the presence of pore and their sizes were performed using a scanning electron microscope Nova FEI Company FEI 450, using a magnification of 700 to 110 000. Samples were observed in the contrast of the secondary electrons (SE).

### 4. RESULTS

The scanning electron microscope (SEM) images of the coal used for our experiment are presented in Figures 5 and 6.



**Fig. 5.** SEM image of selected area of coal sample at magnification:  
a) 400×; b) 800×; c) 1200×



**Fig. 6.** SEM image of selected area of coal sample at magnification:  
a) 600×; b) 800×; c) 1000×

Figure 5 shows BSE image of the selected area of the sample at a magnification of a) 400×, b) 800×, c) 1200×. Figure 6 shows the BSE image of another area of the sample.

Based on the results of laboratory tests it may be noted that the analyzed coal sample has a granular structure, as shown in the figures.

It is also visible that that the structure is stratified, and there is a lot of slots and free space between grains, which can accumulate methane in coal structure.

The field-effect scanning electron microscope (FE-SEM) images of the coal sample used in the experiment are presented in Figures 7–10.

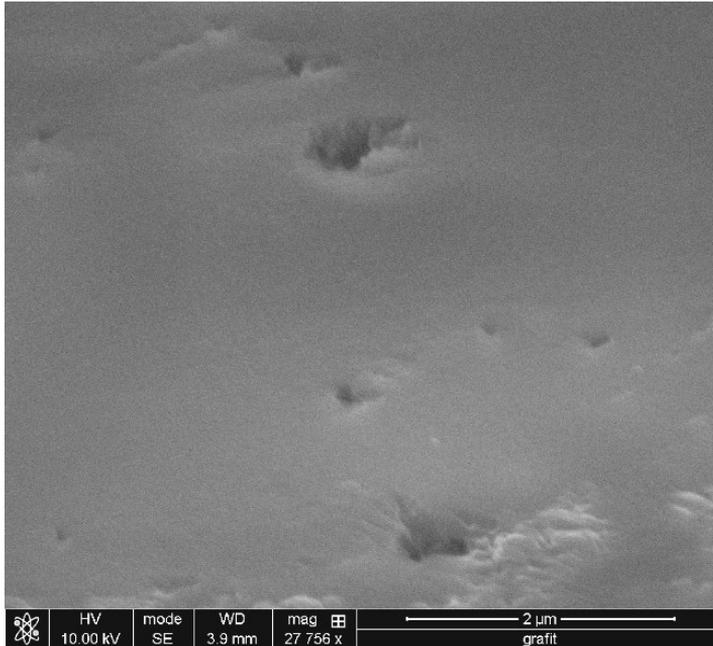
In Figure 9 at a magnification of 83 790x the presence of pores of size of about 500 nm can be seen.

In Figure 7 with the lowest magnification of 27 756x is a picture of a large part of the surface of the sample with a number of pores of different sizes in the range from 100 to 600 nm is presented.

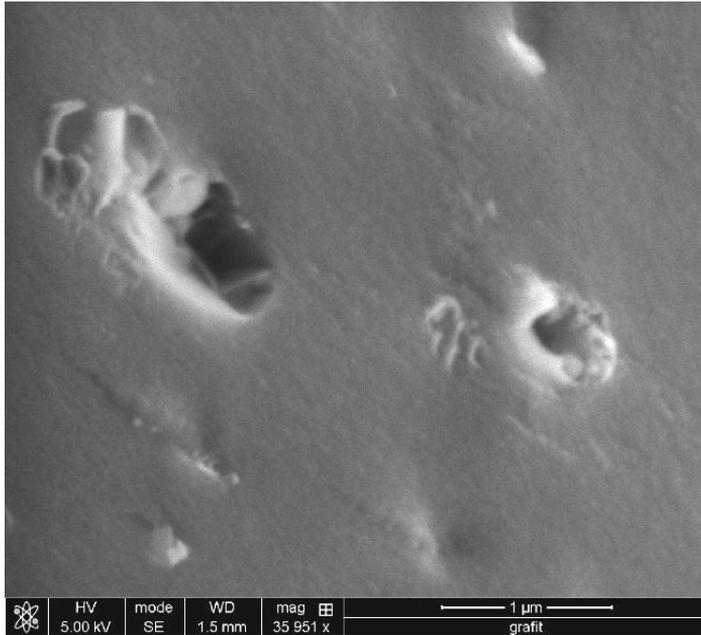
In Figure 8 at a magnification can be observed of 35 951x the image the presence of pores of size from about 250 to 500 nm.

Further analysis showed the presence of pores of different sizes in the coal structure. Porous coal structure is characterized by a high degree of heterogeneity related to the presence of pores of different shapes and sizes.

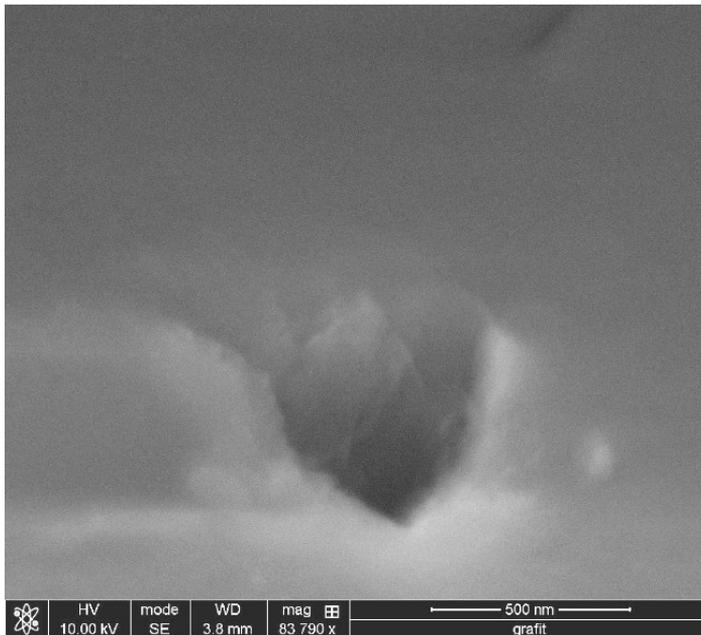
The smallest diameter of the pores of about 62 nm is shown in Figure 10 at a magnification of 111 044x.



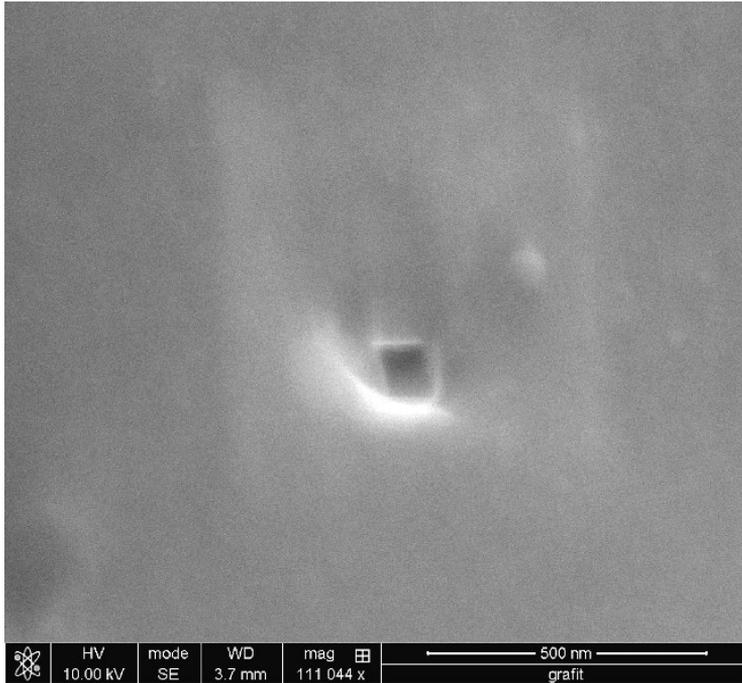
**Fig. 7.** SEM images of the selected surface coal sample at magnification 27 756×



**Fig. 8.** SEM images of the selected surface coal sample at magnification 35 951×



**Fig. 9.** SEM images of the selected surface coal sample at magnification 83 790×



**Fig. 10.** SEM images of the selected surface coal sample at magnification 111 044×

## 5. CONCLUSIONS

1. In the paper was presented the research of the coal surface morphology and the coal porosity, carried out with the use of SEM equipment.
2. It was observed that the coal microstructure is grainy. The large amount of free space between grains can contain methane. Also the grain structure is characterized by large surface area what can cause a big amount of  $\text{CH}_4$  to be adsorbed on the coal grains.
3. The nanoporosity of the coal grains was observed during SEM study. The sizes and shapes of pores are miscellaneous. However the dimension of 62–600 nm allow to draw a conclusion, that the nanopores can contain a few particles of  $\text{CH}_4$  (4 Å), which can be released by the  $\text{CO}_2$  particle (2.54 Å).
4. Finally, on the base of presented research it can be concluded that the method of coal demethanation with the use of  $\text{CO}_2$  can be economically and ecologically effective, and can increase the mine safety.
5. For the demethanation technology development the further experiments – numerical and laboratory ones are planned. The experiments will be consider coal fracturing with the use of  $\text{CO}_2$ . Then the will carried experiment in the real mine environment out.

## REFERENCES

- [1] Enaney B. Mc, Mays T.J.: *Characterization of micropores in carbons, w: Porosity in carbons: Characterization and applications*. Ed J.W. Patrick, E. Arnold, London, 1995.
- [2] Melnichenko Y.B., He L., Sakurovs R., Kholodenko A.L., Blach T., Mastalerz M., Radliński A.P., Cheng G., Mildner D.F.R.: *Accessibility of pores in coal to methane and carbon dioxide*. Fuel, vol. 91, nr 1, 2012, 200–208.
- [3] Gonet A., Nagy S., Rybicki C., Siemek J., Stryczek S., Wiśniowski R.: *Technologia wydobywania metanu z pokładów węgla*. Górnictwo i Geologia, t. 5, z. 3, Kraków 2010, 5–25.
- [4] Bumb A.C. and McKee C.R.: *Gas-well testing in the presence of desorption for coalbed methane and devonian shale*. SPE Formation Evaluation, March 1988.
- [5] Hagoort J.: *Fundamentals of gas reservoir engineering*. Developments in Petroleum Science, vol. 23, Elsevier, Amsterdam, 1988; 328 pp. Dfl. 185.00, ISBN 0-444-42991-3.
- [6] Harpalani S. and Schraufnagel A.R.: *Influence of matrix shrinkage and compressibility on gas production from coalbed methane reservoirs*. Paper SPE 20729, Proceedings of SPE Annual Technical Conference and Exhibition, September 1990.