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STRUCTURAL AND TEXTURAL CHARACTERISTICS OF SELECTED COPPER-BEARING ROCKS AS ONE OF THE ELEMENTS AIDING IN THE ASSESSMENT OF GASOGEODYNAMIC HAZARD

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Abstract: The characteristics of copper-bearing rocks that include the structural and textural parameters are an important factor determining a possible gas accumulation in those rocks. In September 2009, in the Rudna copper mine in Poland, an outburst of gases and dolomite occurred. The analysis of the outburst mass showed that one of the main causes of the outburst was the different structural properties such as high porosity and presence of gas in the pores. This paper presents data from the structural analysis of dolomite from the Polkowice-Sieroszowice copper mine and the Rudna copper mine. Seven rock samples from various areas of the mines were tested by the following methods: mercury porosimetry (MIP), low pressure gas adsorption (LPNA), scanning electron microscopy (SEM), computed microtomography (micro-CT). The SEM analyses of the rock samples allowed pores of various sizes and shapes to the observed. The porosity (MIP) of the dolomite changed in the range of 3–15%. The total micro and mesopore volume (LPNA) was from 0.002 cm^3/g to 0.005 cm^3/g. The macropore volume (MIP) was from 0.01 cm^3/g to 0.06 cm^3/g and the mean macropore diameter was from 0.09 μm to 0.18 μm. The dolomite samples varied in the surface area (LPNA) (0.7–1.5 m^2/g) and the pore distribution. The structure of dolomite determines the possibility of the occurrence of gasogeodynamic phenomena and hence it is urgent that research be conducted into its changeability. To better understand the gasogeodynamic processes in copper-bearing rocks, it is necessary to constantly monitor and analyse in detail those areas that have different structural properties.

Key words: *gasogeodynamic hazard, copper-bearing rocks structure*

1. INTRODUCTION

One of the most serious threats in the mining industry is gasogeodynamic hazard, which includes rock and gas outbursts. A rock and gas outburst is defined as a process of rock crushing and a dynamic movement of the rock and gas mixture into the excavation area (Lama and Bodziony [9], Beamish and Crosdale [2], Cao et al. [5]). According to the majority of theories and scientific research, a rock and gas outburst is triggered mainly by the energy of gases released from a rock mass and a necessary condition for it to occur is the presence of gas pockets in a rock mass (Li et al. [10], Jiang et al. [7]). Additional factors that contribute to the occurrence of an outburst are among others different structural properties of a rock mass in its restricted areas, changes of tensions of a rock mass due to its exploitation as well as a working face approaching faults (Shepherd et al. [15], Xue et al. [18], Lianchong et al. [11], Wierzbicki and Młynarczuk [17]).

Until recently, gasogeodynamic phenomena were associated mainly with coal mines. In recent years, gasogeodynamic hazard has also occurred in the Polish copper mining industry (Butra and Kijewski [4]). In September 2009, in T-169a drift at the depth of 1200 m of the Rudna copper mine, a gas and dolomite outburst occurred, which was the first such incident in the world (Mirek et al. [13], Mirek et al. [12], Wierzbicki and Młynarczuk [17]). It inspired a series of studies of gasogeodynamic hazard in copper mines. The research revealed that one of the causes of the gas and dolomite outburst in Rudna copper mine was the disturbance of gasogeodynamic balance of the rock mass, which was a consequence of drilling a drift by blasting. The disturbances occurred in the area including a previously unidentified layer of dolomite characterised by high porosity, in which gas accumulated under high pressure. The post-outburst cavern covered a few-meter-high dolomite layer and reached the anhydrite floor, which remained intact. The tension of the rock mass resulting from the depth of the mining works as well as the geomechanical properties of the porous dolomites also contributed to the occurrence of the outburst.

After 2009 the incident in the Rudna copper mine, forecasting of gasogeodynamic hazard was introduced. It involved drilling experimental boreholes prior to excavation works. The performed analyses indicated that the presence of gas pockets in the rock mass was directly related to the geological structure of the deposit and the thickness of the dolomite layers lying above copper-bearing slate (Mirek et al. [13]). Typical dolomite has homogeneous, massive and compact structure and is characterised by poorly developed porosity, which in the major part of the deposit is of the order of a few percent. However, the porosity and fractures in the cores from the experimental boreholes falling into the gas pocket was found to be higher up to 165% (Biliński et al. [3]). The analyses of the post-outburst mass from 2009 revealed similarly increased porosity of the dolomite (Wierzbicki and Młynarczuk [17]). Identifying those parts of a deposit that have higher porosity can be one of the methods aiding in the assessment of gasogeodynamic hazard in copper ore mines.

The gasogeodynamic hazard within the copper deposits is related to the geological structure of the southern part of the Fore-Sudetic Monocline. It is a pseudo-layer covering Rotliegent sandstone and Zechstein shales and dolomites (Godyń [6]). The presence of crude oil and natural gas deposits in the vicinity of the Monocline also contributes to the seriousness of the gas threat. The gases occurring in that area can be divided into flammable gases (methane and other hydrocarbons), hydrogen, suffocating gases such as carbon dioxide, nitrogen as well as poisonous gases such as carbon monoxide, sulphur dioxide and hydrogen sulphide. The presence of gases was recorded in all the rock formations that are within the range of mining operations. They can be dissolved or sorbed on the rock surface. They can also occur in the pore space of the rock mass.

Giving priority to health and safety at work in the face of gasogeodynamic hazard justifies the need to make reliable forecasts so that proper preventive steps can be taken. Thus, thorough research into the nature of gasogeodynamic phenomena in copper mines is necessary. Current knowledge and experiences indicate that efficient assessment of possible occurrence of gasogeodynamic phenomena can involve: (i) locating those areas of a rock mass that have different structural and textural properties, (ii) determining the gas content of that part of a rock mass.

In this paper, the authors present the results of the analysis of the structural and textural properties of seven dolomite samples coming from the Legnica-Głogów Copper Belt area. The aim of the research was to estimate the possibilities of locating different parts of rock mass by analysing samples extracted from various parts of the deposit.

2. EXPERIMENTAL METHODS

2.1. SAMPLES

The analyses were performed using dolomite rocks from the Legnica-Głogów Copper Belt area – Polkowice-Sieroszowice copper mine (P1, P2, P3, P4, P5) and Rudna copper mine (P6, P7). The dolomite cores came from various parts of the mines.

2.2. ANALYTICAL TECHNIQUES

For the purpose of the study, the cores were crushed to the grain fractions of 5–30 mm. The porous structure was analysed taking advantage of mercury porosimetry (MIP) and low pressure nitrogen adsorption (LPNA). In order to determine the percentage porosity, the surface area as well as the volume and size of the macropores Thermo Scientific Pascal porosimeter (MIP) was used including a low-pressure module (Pascal 140) – 10^{-5}–10^{-1} MPa and a high-pressure module (Pascal 440) – 0.1–150 MPa. The method consisted in intruding mercury into the open pores of a sample under strictly controlled pressure. It was based on the Washburn (Washburn [16]) equation when measuring the mercury intrusion and on the assumption that size of the pore diameter is proportional to the pressure difference Δp, required for the intrusion of mercury into the pores.

The structural parameters in the range of micro and mesopores were characterised with a surface area analyser ASAP 2020 (Micromeritics) using the low-pressure nitrogen adsorption method (LPNA). Barrett–Joyner–Halenda (BJH) (Barrett et al. [1]) analysis, which is based on the Kelvin equation (Klobes et al. [8]) was used in the calculations.

In BJH method, layers of particles adsorbed on the pore walls are treated as an adsorption film. Due to the phenomenon of capillary condensation, as the pressure increases so does the thickness of the adsorption film. Through determining that thickness it is possible to specify the pore volume and the surface area of the sample. The measurements were made in the absolute pressure range of 0–0.1 MPa, at the temperature of liquid nitrogen (77 K) using nitrogen as the adsorbate.

For observation purposes of dolomite surface, two samples were selected and subjected to scanning analysis on a scanning electron microscope EDAX Apollo XP (SEM-EDS). It is equipped with energy dispersive X-ray spectroscope EDAX Apollo X with secondary electrons SE and backscattered electrons BSE. Secondary electrons have low energy and make it possible to observe phase changes of the surface topography of a sample. Backscattered electrons, due to high energy, allow information of the diversity of sample composition to be obtained. The microscope is also equipped with an energy dispersive X-ray spectroscope EDS, which was used to identify chemical elements composing the rocks.

For the purpose of analysis sections of the samples were polished. The measurements were carried out in the low-pressure mode (10^{-2} Pa) at an accelerating voltage of electron beams 20 kV. The samples were crushed. Before the measurements, thin silver layers (Ag) were dusted onto the samples. The images were recorded with magnifications in the range of ×100–2500. Microanalyses EDS were performed in selected areas of the samples.

Microtomographic analysis (micro-CT) using Benchtop CT160Xi (X-tech Nikon) was performed to obtain quantitative and volumetric characteristic of the pores. The open and closed pores were divided into seven classes according to the space volume they occupied in the rock. The volume unit was voxel, which corresponds to the volume equalling 6 μm × 6 μm. × 6 μm. Dolomite samples were selected for SEM analysis. It consisted in emitting an X-ray, which – passing through the sample – was dispersed and absorbed. The images were taken at the voltage of 75 kV and the intensity of 110 μA. The scanning time of each sample was 2.5 h.

3. RESULTS

To recognise structural and textural properties of the dolomites from the copper mines the following parameters characterising them were identified: percentage porosity, surface area, volume and average pore size. This involved a number of methods such as mercury porosimetry (MIP), low pressure nitrogen adsorption (LPNA) as well as scanning electron microscopy (SEM) and computed microtomography (micro-CT). Pore classification according to IUPAC (Rouquerol et al. [14]) was applied to interpret the results. Due to the fact that the applied methods differed in terms of the measurement specificity, the range of the pore structures that they investigated varied, which is presented in Fig. 1.

3.1. POROSIMETRY ANALYSIS

The MIP analysis showed the dolomites had various structural parameters in the range of macrospores. The most compact were Rocks P1, P2, and P3 from the Polkowice-Sieroszowice copper mine. Their porosity in percent was the lowest of all the samples and amounted to 3.0–3.5%. Similarly, the total pore volume of the macropores was about 0.01 cm^3/g. In dolomites P4, P5 and P6 the percent porosity fell into the range of 4.6–5.0%. Here, the higher porosity was accompanied by an increase of the pore volume up to 0.02 cm^3/g. The most porous was dolomite P7 from the Rudna copper mine with the porosity of nearly 15% and the total volume of the marcopores amounting to 0.06 cm^3/g. This sample had also pores with the largest diameters, which measured 0.18 μm on average. The surface area of all the dolomites was in the range of 0.1–0.5 m^2/g. The structural parameters are presented in Figs. 2 and 3.

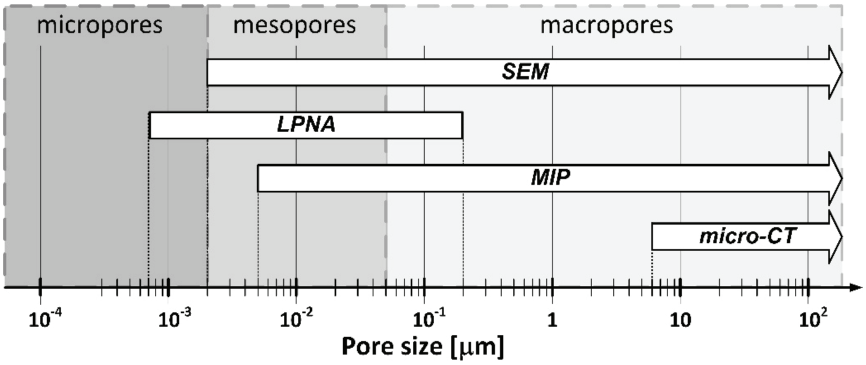

Fig. 1. Determination of pore size distribution

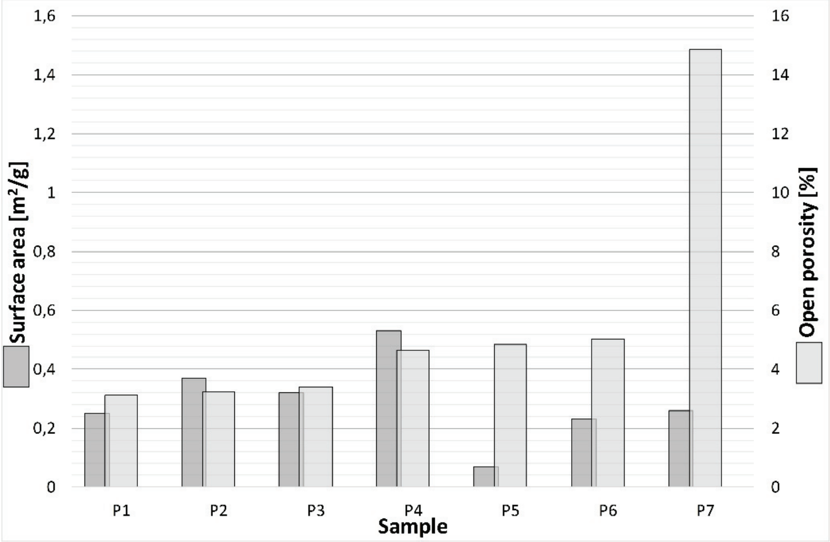

Fig. 2. The percentage porosity and the surface area of the dolomites determined by MIP method

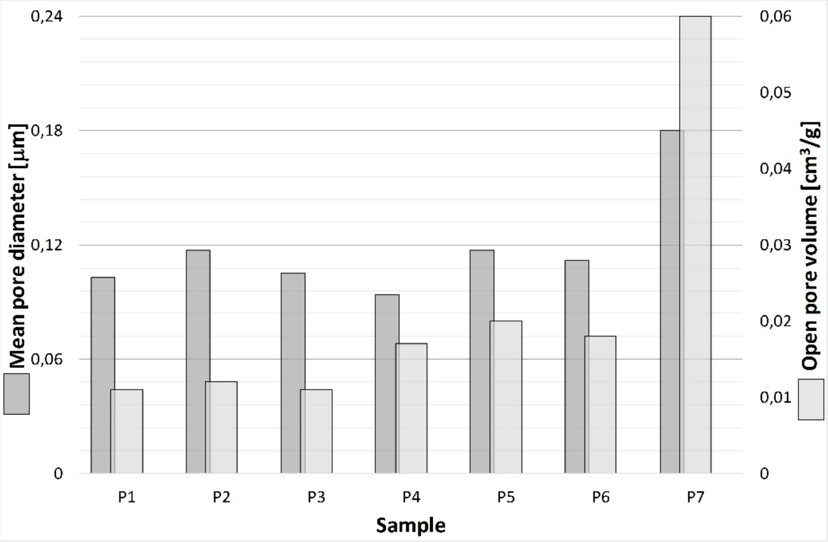

Fig. 3. Total volume and the average size of the pores in the dolomites determined by MIP method

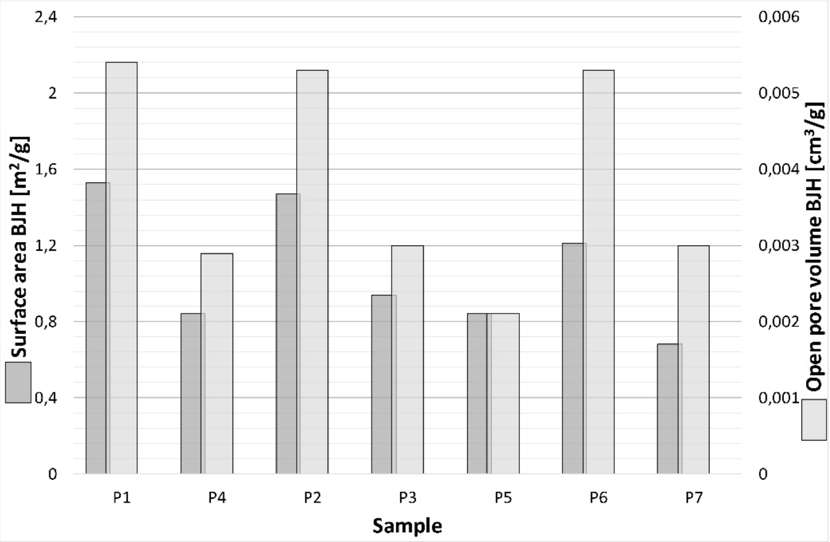

Fig. 4. Total pore volume and the surface area of dolomites determined by LPNA method

The structural parameters, as regards micro and mesopores, were determined by LPNA method. The total volume of the open pores was in the range of 0.002–0.005 cm^3/g and the surface area ranged from 0.7 m^2/g to 1.5 m^2/g. Rocks P1, P3 and P5 had the best developed microstructure as compared to the other rocks, which showed in the largest surface area and pore volume. The other dolomites had lower structural parameters. Dolomite P5 from the Polkowice-Sieroszowice copper mine had the least developed microstructure, the surface area at the level of 0.8 m^2/g and the smallest pore volume (0.002 cm^3/g).

3.2. SEM-EDS MEASUREMENTS

The dolomites from the Polkowice-Sieroszowice copper mine, characterised by extreme levels of porosity, were subjected to SEM analysis. Figure 5 shows an image of the microstructure of the surface of rock P1 obtained using of the BSE detector. The SEM analysis showed that dolomite P1, having the smallest porosity of all the samples (MIP), had massive structure with crystals closely adjacent to one another. A dolomite phase was distinguished in the rock with scattered sulphate minerals and small pores of irregular shapes. The analysis carried out on the edges of the pores revealed the presence of silicon compounds. Also, single cracks filled with a sulphate phase and small clusters with lead sulphite were identified in the rock. EDS analysis was performed for the dolomite phase and sulphate phase. They are presented in Figs. 6 and 7.

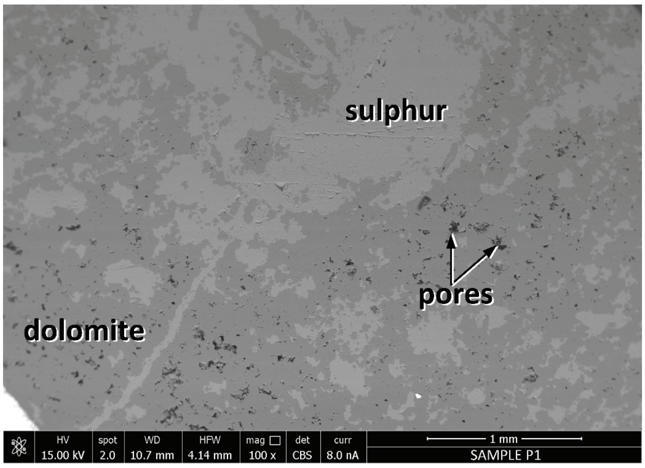

Fig. 5. Image of the microstructure of the surface of dolomite P1, BSE, magnification 100×

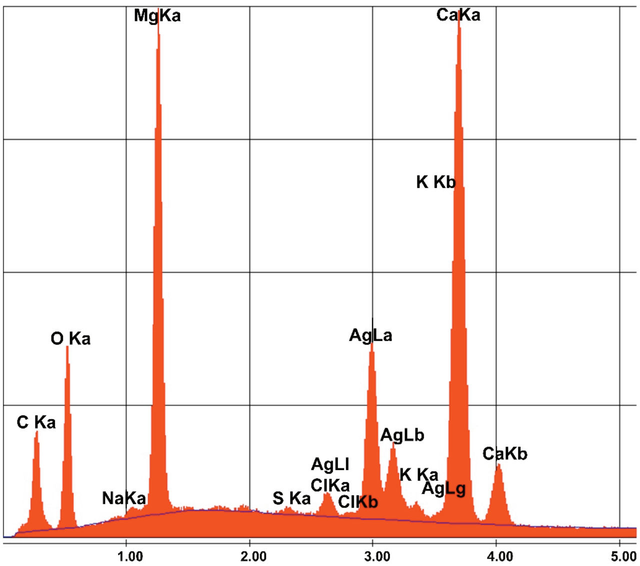

Fig. 6. EDS analysis of dolomite phase in sample P1

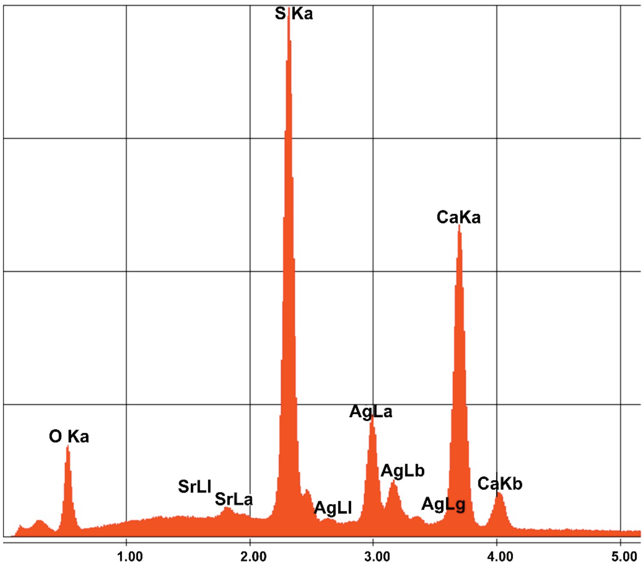

Fig. 7. EDS analysis of sulphate phase in sample P1

Sample P7 from the Rudna copper mine and sample P5 from the Polkowice-Sieroszowice had the highest percent porosity (MIP). The SEM analysis of their surface microstructure revealed that the dolomite phase and the sulphate phase were predominant (Fig. 8, Fig. 9). In both cases, numerous pores can be seen in the dolomite phase. They are small, of irregular shapes and irregularly distributed. Their diameters were in the range from several to several dozen micrometres. Also, concentrations of copper sulphates were observed in small numbers (Fig. 10) and large pores filled with well visible dolomite crystals of rhombohedral habit (Fig. 11). Porous rocks P7 and P5 were less chemically diverse than nonporous rock P1. The copper sulphates as well as two prevailing phases, whose EDS analyses are presented in Figs. 12 and 13, were observed.

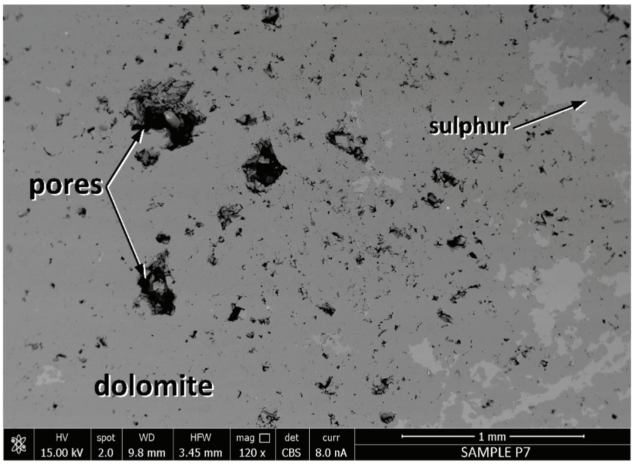

Fig. 8. Image of the microstructure of the surface of dolomite P7, BSE, magnification 120×

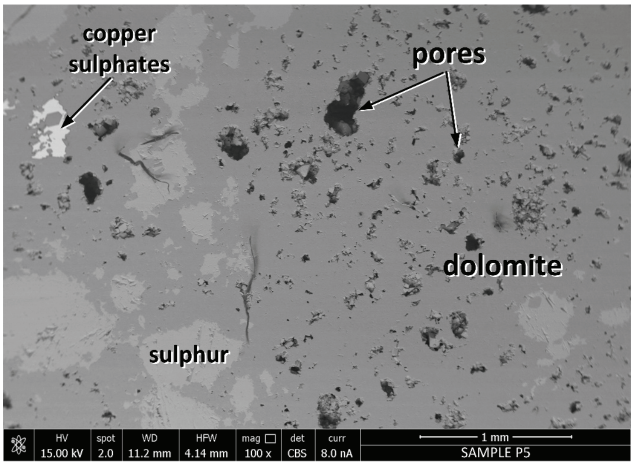

Fig. 9. Image of the microstructure of the surface of dolomite P5, BSE, magnification 100×

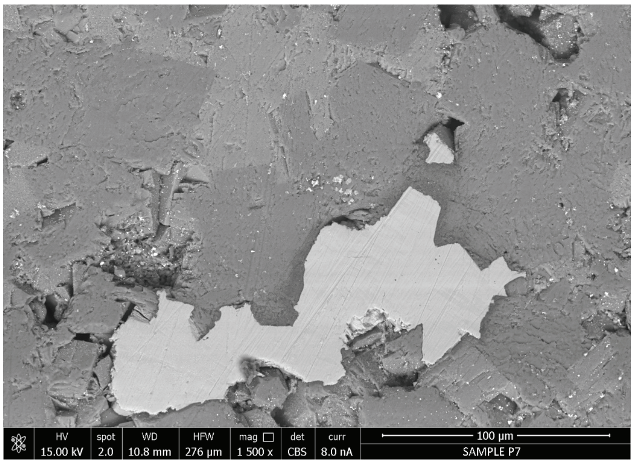

Fig. 10. Image of concentrations of copper sulphates in dolomite P7, BSE, magnification 1500×

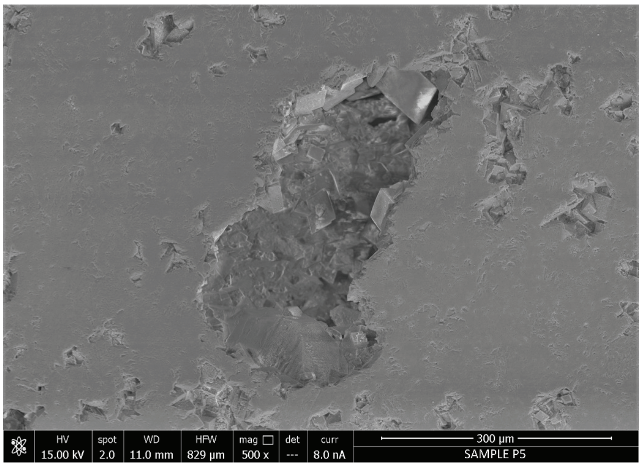

Fig. 11. Images of the pore surface of dolomite P5, BSE and SE image (MIX), magnification 500×

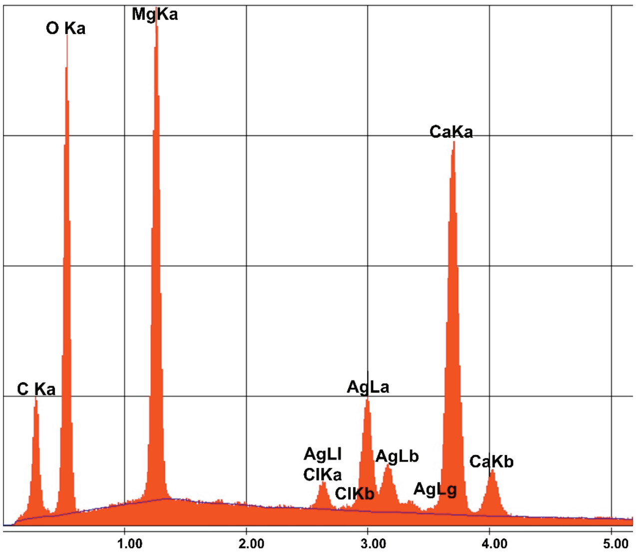

Fig. 12. EDS analysis of dolomite phase in sample P5

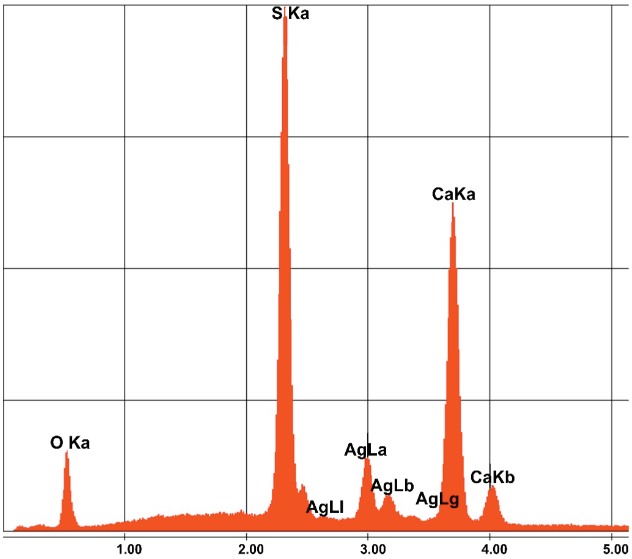

Fig. 13. EDS analysis of the sulphate phase in sample P5

3.3. COMPUTED MICROTOMOGRAPHY

The rocks studied by SEM method (P1, P5) were subjected to computed microtomography (micro-CT). Low-porosity dolomite P1 (MIP) had mostly small volume pores below 9 voxels and was categorised as class 1. The pores from class 3 and class 4 had the biggest share of the pore space in this dolomite. Dolomite P5, with well developed porosity, had mostly pores in the range from 9 to 999 voxels and was categorised as class 1, 2 and 3. These pores, together with class 4 pores, made up the major part of the pore space of that dolomite and their total volume was over ten times that of the pores in sample P1.

Table 1 The pore structure of dolomite P1 and P5 (micro-CT analyses)

Class number	Class volume	Dolomite P1		Dolomite P5	
		Number of pores in class	Class volume	Number of pores in class	Class volume
Unit		voxel	voxel	voxel	voxel
1	<9	120 561	253 166	46 172	331 994
2	10–99	10 187	267 945	86 194	2 534 441
3	100–999	1703	495 047	20 458	5 855 483
4	1000–9999	216	508 153	2540	5 785 282
5	10 000–99 999	2	27 988	70	1 352 927
6	100 000–399 999	0	0	2	313 214
7	>400 000	0	0	0	0

4. CONCLUSIONS

All over the world gasogeodynamic phenomena were a rare event. In recent years, the hazards due to the presence of gas in deposits emerged in the Legnica-Głogów Copper Belt, Poland. This poses a serious problem because gasogeodynamic phenomena in dolomite rocks have so far been hardly investigated. Some of the sparse, available research data of dolomite samples from the spot in Rudna copper mine where a dolomite and rock outburst took place were presented by (Wierzbicki and Młynarczuk [17]). Their analyses revealed that one of the causes of the gas and dolomite outburst was the presence of gas in the pores of the rock and its different structural properties. They concluded that it is extremely important to locate areas of a rock mass which are characterised by higher porosity and increased pore volume. The studies of the post-outburst mass of the dolomite from 2009 from the Rudna copper mine showed that its average porosity was 16% while the porosity of the dolomite samples extracted from the place some distance away was 8%. Optical microscopic analysis also revealed a large number of macropores in the post-outburst mass as compared to the other samples.

The aim of the present research into the dolomites from the Legnica-Głogów Copper Belt was to estimate the possibilities of locating structurally different areas of a rock mass according to structural and textural characteristics of samples from various spots of the mine. Use was made of porosimetric methods (MIP, LPNA), scanning electron microscopy (SEM) and computed microtomography (micro-CT).

The porosimetric analyses allowed the structure of the rocks to be determined taking into account the full range of their porosity. With the MIP method it was possible to characterise the macropores. The LPNA method allowed the analysis of the smallest pores to be carried out and the BJH model used in the calculations made it possible to characterise the mesopores in which capillary condensation takes place. The SEM-EDS analyses allowed the surface topography of the rocks to be observed and their chemical composition to be determined. The number and volume of the open and closed pores in the rock were specified by the micro-CT analysis.

The lowest porosity and the smallest total pore volume were identified by MIP method in dolomite P1 from the Polkowice-Sieroszowice copper mine. As the SEM-EDS analysis revealed, it had massive structure and small pores of irregular shapes occurring between crystals closely adjacent to one another. Apart from the dolomite, also a sulphate phase and concentrations of lead sulphide were observed. Dolomite P1 was a typically fine-pored rock, which was confirmed by the LPNA analyses. They showed that in the range of the smallest pores, it had the largest surface area and the biggest volume compared to the other samples. According to the micro-CT, the smallest volume pores predominated here.

Typically fine-pored structure was also observed in rock P3. The macroporosity and macropore volume of this sample (MIP) had some of the smallest values, whereas the parameters of the micro and mesopores (LPNA) reached the highest level.

Dolomite P5 had the largest macroporosity of all the samples from Polkowice-Sieroszowice. The pores on its surface had the longest mean diameter and the biggest total volume (MIP). The SEM analysis shoved that the pores had diameters up to dozens of micrometres, were irregularly distributed, had irregular shapes and contained well visible crystals of rhombohedral habit. This dolomite was less chemically diverse than dolomite P1 and apart from the sulphate phase, rare concentrations of copper sulphates were also observed.

Of all the rocks the most developed pore structure was detected in dolomites P7 from the Rudna copper mine. The MIP analyses showed that they had the highest percent macoporosity and the biggest macropore volume. Dolomite P7 was characterized by particularly different structural properties, its porosity amounted to above 15% and the pore volume was 0.06 cm^3/g. Also, P7 had bigger pores than the other rocks (MIP) and poorly developed micro and mesoporosity (LPNA).

Comparing the results obtained from the analysis of the dolomites with the data from the post-outburst mass from the Rudna copper mine, it is possible to classify the samples under study as either less or more prone to gasogeodynamic phenomena. Based on the analysis of the porous structure, dolomites P1, P2 and P3 should be categorised as less prone to such phenomena. The values of the structural parameters of dolomites P4, P5 and P6 were found to be higher. However, they were much lower than those of the reference samples. Since dolomite P7 is the most structurally different rock, the area from which it was extracted should be regarded as potentially prone to gasogeodynamic phenomena.

The comparison of structural and textural parameters of the copper-bearing rocks with the dolomites from the outburst area proposed in this paper is an important element that can help to understand the nature of the gasogeodynamic phenomena in dolomite rock in copper mines, and which can aid in the assessment of gasogeodynamic hazard. The identified structural parameters help to localise areas characterised by different structural properties but they do not provide a clear indication of a gasogeodynamic threat. In order to brouden our knowledge on the subject matter, it is necessary to supplement the studies with gas analysis, which will be the aim of the authors in future research.

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