



Textural transformation of thermally treated anthracites

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Abstract

The organic matter of anthracites consists of turbostratic (two-dimensional crystalline order) stacks of three to five aromatic layers called the Basic Structural Units (BSU). The arrangement of BSU forms the texture of anthracites, which corresponds to their chemical and physical properties including optical properties (reflectance values). Thermal treatment leads to the changes in the arrangement of BSU. The character of structural and textural transformation of anthracites during heating can be determined based on the changes of reflectance values of anthracites.

Several anthracites of various ranks were thermally treated over the temperature range of 400 (673 K)–1000 °C (1273 K) in an inert atmosphere (N₂), at atmospheric pressure. The reflectance indicating surfaces (RIS) were reconstructed and reflectance indicatrix parameters were calculated according to the Kilby method specially modified for the study of anthracites.

It was found that the relative increase of reflectance values (R_{\max} , R_{\min}) was greater for the lower metamorphosed samples than for the higher ones. However, the higher rank of initial samples, the greater their final reflectance values and the anisotropy of the texture attained at 1000 °C. It indicates that the texture of the higher metamorphosed anthracites (meta-anthracites) is more flexible for rearrangement during heating, than the texture of the lower metamorphosed anthracites. Moreover, the difference in the changes of reflectance values versus temperature for the higher and for the lower rank anthracites suggests that meta-anthracites have undergone stronger natural metamorphism equivalent to about 200 °C in comparison with the other anthracites.

It could be said that the character of textural transformations of individual anthracites results from the degree of their metamorphism and, in a consequence, from the texture of parent sediments.

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Keywords: Anthracite; Reflectance; Anisotropy; Texture; Thermal treatment

1. Introduction

Anthracites are the high rank coal variety with a specific multiscale organization; structure, microtexture and texture. The structure consists of turbostratic stacks of three to five aromatic layers called the Basic

Structural Units (BSU) (Bonijoly et al., 1982; Oberlin, 1984; Rouzaud and Oberlin, 1990). Polyaromatic stacks are arranged in aggregates (domains) with different dimensions and spatial orientation which form the microtexture of anthracites. Bonijoly et al. (1982) proposed the model of the structure and the microtexture of carbonaceous materials, which well illustrates the multiscale organization of anthracites. According to this model, the microtexture of anthracites is characterized by flattened pores which walls are consisted of the domains. Metamorphism agents

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like lithostatic pressure and oriented strains cause that domains show statistical orientation and form the texture of anthracites. Dimensions of the domains increase together with the degree of metamorphism of anthracites. Simultaneously, the orientation of the BUS and/or domains develops along a preferential plane, most often parallel to the bedding, and the anisotropy of the texture appears (Oberlin and Terriere, 1975; Bonijoly et al., 1982; Oberlin, 1984; Deurbergue et al., 1987; Rouzaud et al., 1999). In addition, Blanche et al. (1995) and Duber et al. (2000) stated that anthracites are heterogeneous materials where several types of multiscale organizations could coexist together.

It is known that the multiscale arrangement of anthracites is closely related to their specific physical and chemical properties. Thus, it could be supposed that the variability in properties of carbon materials and anthracites may be explained by the differences in their texture, i.e. various spatial arrangements of the BSU (Rouzaud and Oberlin, 1989). Thermal treatment of anthracites leads to the changes in their texture that affects their physical and chemical properties and determines the way of their utilization. It seems that the determination of the character of textural transformation of anthracites during heating could be interesting and important from the practical point of view.

The texture of anthracites can be determined by studying their optical properties and the manner of rearrangement of anthracites texture during heating corresponds to the changes of their reflectance values.

Up to now, the most popular way to show the effect of thermal treatment on chemical structure and the texture of coals or carbon materials was to describe the size and shape of optical texture of final products (Patrick et al., 1973; Forrest and Marsh, 1977; Patrick et al., 1989; Marsh and Menendez, 1989; Patrick and Walker, 1991). This method is rather subjective and the results are not unambiguous and difficult to compare. Some authors started to use the measurements of reflectance values to estimate the effect of temperature or pressure on the texture and, in a consequence, on properties of coals or coal products (Goodarzi, 1985; Goodarzi and Murchison, 1973; Bustin et al., 1986; Morishita et al., 1986; Wilks et al., 1993; Bustin et al., 1995; Alonso et al., 2001). In

this case, problems with the heterogeneity of optical properties of studied samples could occur.

At the end of the 1980s, Kilby (1988, 1991) proposed the method for determining the optical character of bituminous coals based on the measurements of maximum and minimum reflectance values. This method makes it possible to define optical properties of powdered samples of coals and to express them numerically (unambiguously). Essential modification of Kilby's method, to use it for the study of the chemical structure and the texture of heterogeneous samples such as anthracites or carbon materials, has been done and described (Duber et al., 2000). It seems that the method mentioned above defines optical properties of coals and carbon products better than previous ones and allows for more detailed study of the character of structural and textural transformations during technological processes.

The aim of this work was to determine the character of structural and textural transformations of anthracites during thermal treatment up to 1000 °C on the basis of reflectance values of anthracites using the modified Kilby's method (Duber et al., 2000). Detailed description of the methods used in our study is given in Section 2.

2. Experimental methods

2.1. Sampling

Six anthracites of various rank: Moszczenica and Walbrzych from Poland, Donetsk and Svierdlovsk from Ukraine, La Mure from France and Listvianski from Russia were heated at the temperature range of 400 (673 K)–1000 °C (1273 K) in an inert atmosphere (N₂), at atmospheric pressure, with a heating rate of 4°/min. In the final temperatures, samples were kept 1 h and then cooled in inert atmosphere.

Geographical origin of anthracites studied is presented in Table 1. Chemical and petrographical parameters and mean reflectance values of the raw anthracites are shown in Table 2. Detailed petrographical characteristics of anthracites studied were presented earlier (Duber et al., 2000).

It can be supposed, from the point of view of chemical composition and mean reflectance values,

Table 1
Geographical origin of the samples studied

Sample	Country	Coal basin (region or mine)
Walbrzych	Poland	Lower Silesian Coal Basin (Walbrzych mine)
Moszczenica	Poland	Upper Silesian Coal Basin (Moszczenica mine)
Donetsk	Ukraine	Donbass
La Mure	France	La Mure
Svierdlovsk	Ukraine	Donbass (Svierdlovsk region)
Listvianski	Russia	Kuzbass

that Donetsk, Svierdlovsk, La Mure and Listvianski anthracites underwent a strong degree of metamorphism and, according to German classification, they might be called meta-anthracites (Stach et al., 1982). Two other anthracites: Moszczenica and Walbrzych represent weaker stages of coalification.

2.2. The method of calculation and analysis of optical parameters of the samples studied

The maximum and minimum reflectance values were measured for all raw and thermally treated samples. Reflectance measurements were made at room temperature, with oil immersion, with a reflected light microscope (Axioskop MPM 200-Opton, Germany), using monochromatic linearly polarized light of $\lambda = 546$ nm. Measurement points, 200–300 per sample, were carried out. On the basis of the data collected, principle axes of the reflectance indicating surface, RIS (R_{MAX} , R_{INT} , R_{MIN}) and characteristic

Table 3
Explanation of reflectance (R) symbols used in the paper

Symbol	Explanation of the symbol
RIS	Reflectance indicating surface
R_{MAX}	Maximum reflectance axis of RIS
R_{INT}	Intermediate reflectance axis of RIS
R_{MIN}	Minimum reflectance axis of RIS
R_{st}	RIS style (parameter of RIS)
R_{ev}	Reflectance of equivalent volume isotropic RIS (parameter of RIS)
R_{am}	RIS anisotropy magnitude (parameter of RIS)
R'_{min}	Apparent minimum reflectance value (measured)
R_{min}	Mean apparent minimum reflectance value (mean of measurements)
R'_{max}	Apparent maximum reflectance value (measured)
R_{max}	Mean apparent maximum reflectance value (mean of measurements)
R_m^o	Mean reflectance value (mean of measurements)

RIS parameters (R_{ev} , R_{st} and R_{am}) (see Table 3) were calculated by the Kilby method (Kilby, 1988, 1991) which was specially modified for anthracites (Duber et al., 2000). The RIS reconstruction for all samples was carried out to determine the differences in textural transformations during heating of anthracites.

2.2.1. Kilby's method

Kilby (1988, 1991) proposed the method for determining the RIS of coal based on the measurements of apparent R'_{max} and apparent R'_{min} (Table 3) reflectance values on cross-sections of non-oriented coal grains. “Apparent”, because only in the case when the grain cross-section contains the main axis

Table 2
Chemical and petrographical compositions and mean reflectance values of anthracites studied

Sample	Chemical composition [wt.%]					Petrographical composition [vol.%]				R_m^o [%]
	A ^d	C ^{daf}	H ^{daf}	(O+N) ^{daf}	V ^{daf}	Vt	I	L	MM	
Walbrzych	8.9	90.4	3.8	5.1	9.2	81.2	2.8	0.0	16.0	2.11
Moszczenica	4.2	92.3	2.8	4.4	5.4	88.7	11.2	0.0	0.1	3.10
Donetsk	2.8	94.4	1.9	3.3	3.1	82.5	11.1	0.0	6.4	4.73
La Mure	6.7	94.5	1.5	1.5	3.4	83.8	15.4	0.0	0.8	4.91
Svierdlovsk	4.9	95.1	1.3	2.7	1.9	88.8	7.8	0.8	2.6	5.07
Listvianski	3.3	98.1	0.3	1.5	1.3	80.5	18.3	0.0	1.2	6.80

A^d—ash content, dry basis; C^{daf}—carbon content, dry, ash free basis; H^{daf}—hydrogen content, dry, ash free basis; (O+N)^{daf}—oxygen and nitrogen content, dry, ash free basis; V^{daf}—volatile matter content, dry, ash free basis; Vt—vitrinite content; L—liptinite content; I—inertrinite content; MM—mineral matter content; R_m^o —mean reflectance value (in oil immersion).

R_{MAX} and/or R_{MIN} of the RIS, the measured R'_{max} and/or R'_{min} values correspond to real R_{max} and/or R_{min} (Table 3) reflectance values of coal studied. In other cases, $R_{MAX} > R'_{max} > R_{INT}$ and $R_{INT} > R'_{min} > R_{MIN}$. Three principal axes of RIS are determined by means of a graphical method which consists of plotting measured R'_{max} and R'_{min} reflectance values (axis of abscissae) versus bireflectivity ($R'_{max} - R'_{min}$) (axis of ordinates) in data point of measurement. Minimum value in R'_{min} data set on Kilby's cross-plot is

considered as R_{MIN} axis and maximum value in R'_{max} data set as R_{MAX} axis of RIS. The border between R'_{min} and R'_{max} data sets means the requested R_{INT} axis.

The shape and dimensions of a RIS which characterize the texture of coal (degree of order and anisotropy) and chemical structure of BSU of coal are unambiguously determined in orthogonal co-ordinate system by means of R_{MAX} , R_{INT} and R_{MIN} values. To improve the definition of a RIS, Kilby introduced

Table 4
Optical parameters (measured and calculated) of initial and thermally treated anthracites

Sample		Measured values [%]		Calculated values					
		R_{max}	R_{min}	Axes of RIS			Parameters of RIS		
				R_{MAX}	R_{INT}	R_{MIN}	R_{ev}	R_{st}	R_{am}
Walbrzych	raw	2.30	1.95	2.38	2.25	1.58	2.04	-21.29	0.080
	400	2.34	1.99	2.45	2.27	1.64	2.09	-17.79	0.077
	600	3.46	3.10	3.68	3.48	1.82	2.86	-24.38	0.131
	800	6.21	5.12	6.33	6.11	2.97	4.86	-26.65	0.141
	1000	6.71	5.44	6.86	6.53	4.05	5.66	-23.83	0.102
Moszczenica	raw	3.33	2.89	3.46	3.20	2.54	3.04	-14.09	0.060
	400	3.59	3.13	3.69	3.50	2.68	3.26	-19.71	0.063
	600	4.25	3.69	4.47	4.10	3.15	3.86	-14.54	0.067
	800	6.42	5.43	6.75	6.25	4.42	5.71	-18.85	0.081
	1000	7.67	6.39	8.04	7.46	5.12	6.75	-19.33	0.087
Donetsk	raw	6.19	3.69	6.47	5.98	2.08	4.32	-24.16	0.191
	400	6.21	3.93	6.39	6.03	2.40	4.52	-25.31	0.172
	600	6.31	4.02	6.61	6.06	2.27	4.50	-23.35	0.183
	800	7.40	4.92	7.62	7.18	2.37	5.06	-25.69	0.196
	1000	8.88	6.17	9.15	8.55	3.58	6.54	-24.29	0.166
La Mure	raw	6.14	3.98	6.36	5.92	2.46	4.52	-24.09	0.167
	400	6.22	4.20	6.49	6.02	2.80	4.78	-23.29	0.151
	600	6.47	4.50	6.80	6.21	2.81	4.91	-22.19	0.157
	800	7.05	4.87	7.31	6.78	3.27	5.45	-23.07	0.146
	1000	7.91	5.09	8.27	7.56	3.27	5.88	-22.34	0.162
Sviardlovsk	raw	6.60	3.99	6.91	6.34	2.21	4.59	-23.62	0.192
	400	6.57	3.82	6.86	6.33	2.23	4.59	-24.03	0.190
	600	6.77	4.02	7.12	6.48	2.30	4.73	-22.98	0.190
	800	7.15	4.29	7.52	6.89	2.46	5.03	-23.43	0.189
	1000	8.12	5.04	8.49	7.79	2.80	5.70	-23.47	0.188
Listvianski	raw	8.87	5.33	9.42	8.38	2.90	6.12	-21.46	0.195
	400	8.73	5.98	9.16	8.16	2.87	5.99	-21.49	0.193
	600	8.60	5.72	9.30	8.03	1.83	5.15	-20.86	0.241
	800	8.92	5.57	9.43	8.41	2.75	6.02	-21.85	0.202
	1000	8.83	5.56	9.28	8.34	2.12	5.47	-23.06	0.228

three indicatrix parameters R_{ev} , R_{st} and R_{am} presented by the following equations:

$$R_{st} = 30 - \arctan(a/b)$$

$$R_{am} = (a^2 + b^2)^{1/2}$$

$$R_{ev} = (R_{MAX}R_{INT}R_{MIN})^{1/3}$$

where:

$$a = [1/3 - R_{MIN}/(R_{MAX} + R_{INT} + R_{MIN})] / \cos(30) - b \tan(30);$$

$$b = [R_{MAX}/(R_{MAX} + R_{INT} + R_{MIN}) - 1/3]$$

The R_{st} parameter determines optical character (style) of RIS and its value varies from -30 to $+30$. When the R_{st} is ± 30 , then the optical character of a RIS is uniaxial positive (+) or negative (-). Values between -30 and $+30$ indicate biaxial positive or negative character of the texture. When the value of R_{st} is 0, the character of RIS is biaxial even and then $R_{INT} = (R_{MAX} + R_{MIN})/2$.

The R_{am} parameter characterizes RIS anisotropy and informs about the elongation of the RIS shape. R_{am} is 0 when $R_{MAX} = R_{INT} = R_{MIN}$ (sphere) and the character of the texture is isotropic.

The R_{ev} parameter (RIS equivalent volume) is the reflectance value of an isotropic RIS with a volume equal to the given RIS. It is related to the chemical structure of BSU of sample studied (Duber et al., 2000).

2.2.2. Modification of Kilby's method

On the basis of computer simulations, Kilby demonstrated that R'_{max} and R'_{min} data sets on the cross-plot are separable and their borderline corresponds to R_{INT} axis value of the given RIS of studied material. In the case of anthracites, R'_{max} and R'_{min} data sets overlap one another, which makes it impossible to get an accurate determination of R_{INT} value. Previous study of anthracites from various places in the world revealed that the degree of overlapping of experimental data set R'_{max} and R'_{min} is the effect of the heterogeneity of the structure and texture of anthracites (Duber et al., 2000). It was shown that each Kilby cross-plot is the sum of elemental diagrams, which represent different textural classes (different types of the BSU and domains arrangement) of studied anthracite. Reflectance values (main axes of the RIS) characterizing general structure and texture

of anthracites were calculated as weighted means values which regard the proportion of each textural class within the individual anthracites.

3. Results and discussion

The results of measurements and calculations of optical parameters of anthracites studied are shown in Table 4. It was found that the maximum (R_{max}) and the minimum (R_{min}) reflectance values increase with increasing temperature for all samples studied. The increases are a consequence of devolatilization, increasing aromatization and ordering of the aromatic lamellae (Van Krevelen, 1993), which in turn, affect changes in the porosity of anthracites (quantity, dimensions and shapes), another influencing factor for R_{max} and R_{min} values (Duber and Rouzaud, 1999). Fig. 1 shows that the lower the rank of parent anthracites, the greater the relative increase of their reflectance values during heating.

For meta-anthracites Donetsk, La Mure and Svierdlovsk, the changes of reflectance values are insignificant below $600\text{ }^{\circ}\text{C}$, while in the case of Moszczenica and Walbrzych anthracites strong

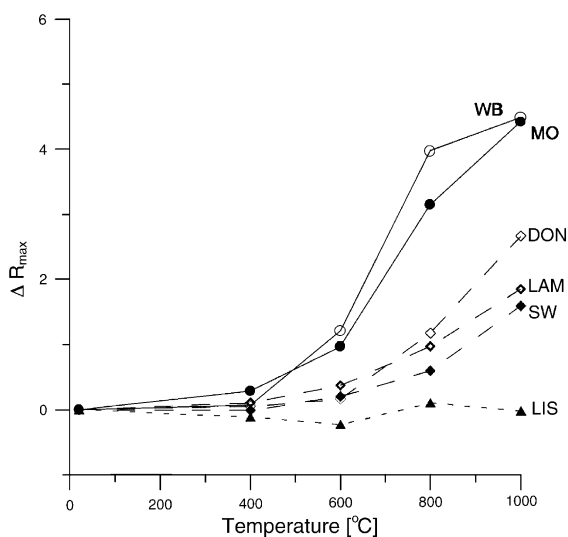


Fig. 1. The increase of delta R_{max} values versus the temperature of treatment [$\Delta R_{max}(T_{i+1}) = R_{max}(T_{i+1}) - R_{max}(T_i)$]: WB—Walbrzych, MO—Moszczenica, DON—Donetsk, LAM—La Mure, SW—Svierdlovsk, LIS—Listvianski.

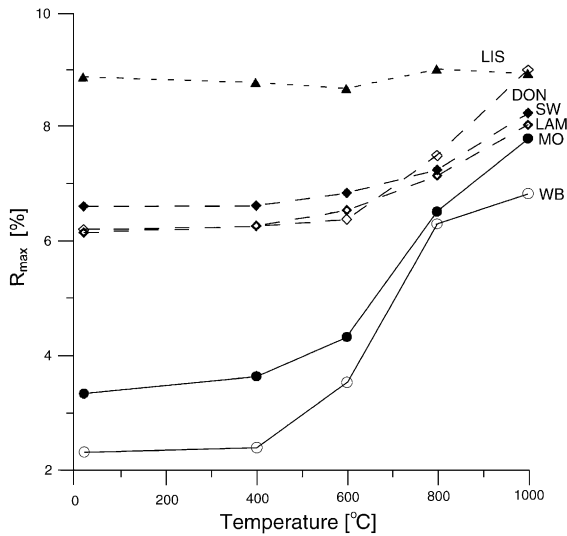


Fig. 2. The changes of R_{\max} values versus the temperature of treatment: WB—Walbrzych, MO—Moszczenica, DON—Donetsk, LAM—La Mure, SW—Svierdlovsk, LIS—Listvianski.

changes of reflectance values occur at 400 °C. Perhaps, prior to laboratory heating, Donetsk, La Mure and Svierdlovsk anthracites had undergone higher degree of natural metamorphism equivalent to about 200 °C than Moszczenica and Walbrzych anthracites. However, the effect of other metamorphism agent, such as pressure or shears, should also be taken into consideration (Bustin et al., 1995). Sample Listvianski does not reveal any changes of reflectance values even at 1000 °C (Fig. 2). The degree of natural metamorphism of Listvianski anthracite is the strongest among all anthracites studied, which would partially explain the minimal changes of its reflectance values during laboratory heating. It was also stated that its texture is extremely heterogeneous (Duber et al., 2000).

R_{\max} and R_{\min} values have a direct effect on R_{ev} which characterizes chemical structure of BSU of anthracites (Duber et al., 2000). An increase in R_{ev} together with the temperature is a measure of BSU structural order with increasing temperature. Fig. 3 illustrates that the lower the rank of raw anthracite, the higher the increase of R_{ev} value and thus, the greater change of chemical structure of BSU. The value of R_{ev} hardly increases for Listvianski anthracite which may indicate that its natural metamorphism occurred under

conditions comparable to those obtained in the laboratory at 1000 °C. This means that the chemical structure of BSU of Walbrzych, Moszczenica, Donetsk, La Mure and Svierdlovsk anthracites became similar to that of Listvianski anthracite after their treatment at 1000 °C.

R_{st} and R_{am} do not depend on the chemical structure of anthracites (Kilby, 1988, 1991; Duber et al., 2000). They are assumed to be due to three-dimensional arrangement of BSU, i.e. to anthracites texture. We did not observe the correlation between R_{st} parameter value describing optical characteristic of anthracites and the temperature of their treatment. The value of R_{st} for all samples studied indicates that their texture remained biaxial negative.

The R_{am} values show that each anthracite retains some individuality in textural alterations during thermal treatment (Fig. 4a–f). There is a clear relation between the degree of the texture anisotropy, expressed by the R_{am} value, and the rank of the parent anthracites. The higher the rank of initial sample, the greater its texture anisotropy after heating at 1000 °C. Finally, anisotropy of the texture of meta-anthracites (Donetsk, La Mure, Svierdlovsk and Listvianski) is

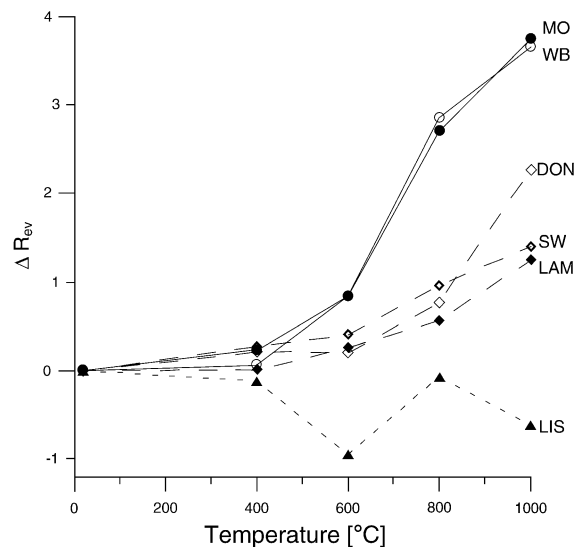


Fig. 3. The increase of delta R_{ev} parameter values versus the temperature of treatment [$\Delta R_{\text{ev}}(T_{i+1}) = R_{\text{ev}}(T_{i+1}) - R_{\text{ev}}(T_i)$]: WB—Walbrzych, MO—Moszczenica, DON—Donetsk, LAM—La Mure, SW—Svierdlovsk, LIS—Listvianski.

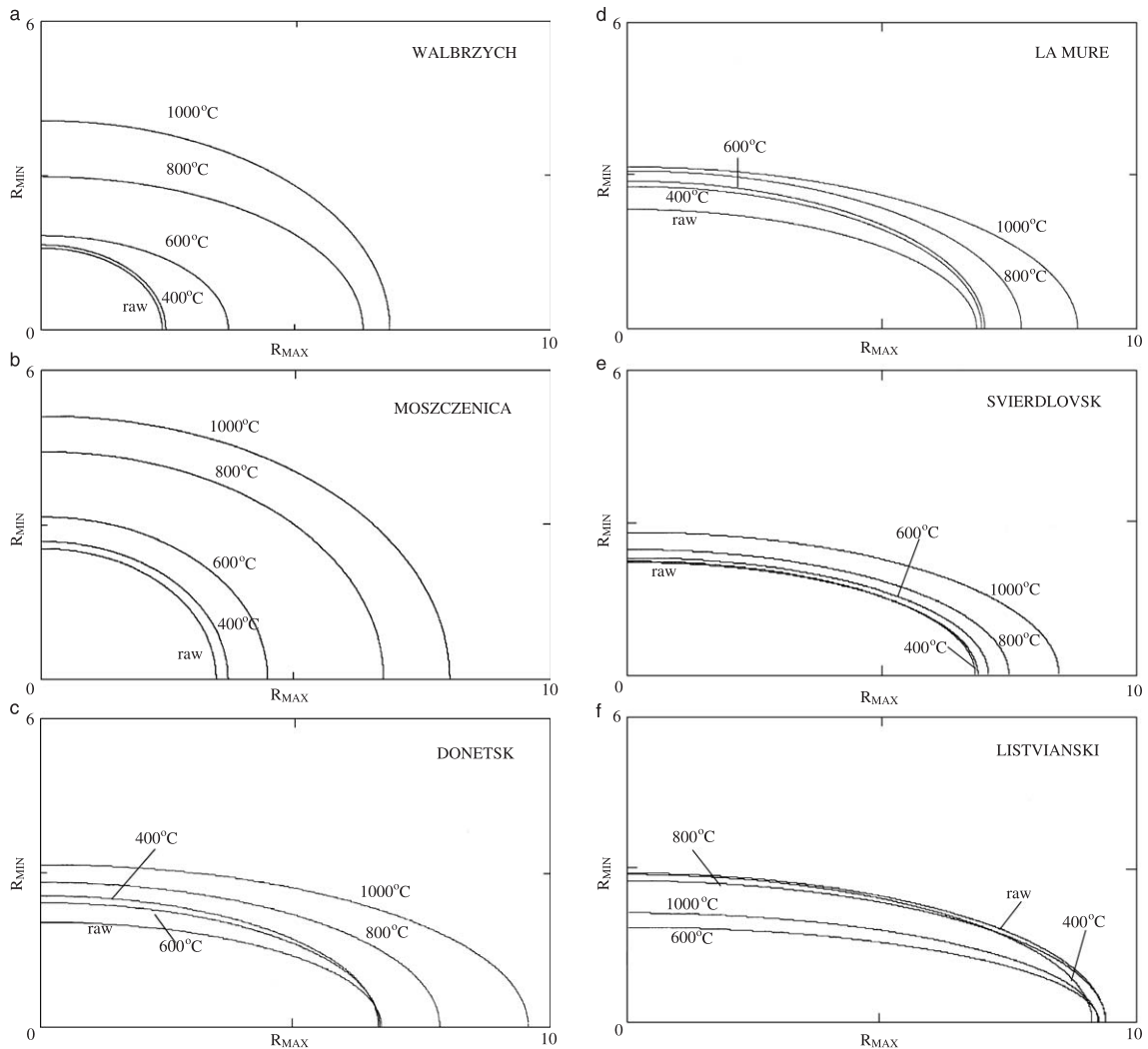


Fig. 4. (a) The cross-sections of the RIS along the principle axes R_{MAX} and R_{MIN} of Walbrzych anthracite thermally treated at various temperatures (400, 600, 800 and 1000 °C). (b) The cross-sections of the RIS along the principle axes R_{MAX} and R_{MIN} of Moszczenica anthracite thermally treated at various temperatures (400, 600, 800 and 1000 °C). (c) The cross-sections of the RIS along the principle axes R_{MAX} and R_{MIN} of Donetsk anthracite thermally treated at various temperatures (400, 600, 800 and 1000 °C). (d) The cross-sections of the RIS along the principle axes R_{MAX} and R_{MIN} of La Mure anthracite thermally treated at various temperatures (400, 600, 800 and 1000 °C). (e) The cross-sections of the RIS along the principle axes R_{MAX} and R_{MIN} of Svierdlovsk anthracite thermally treated at various temperatures (400, 600, 800 and 1000 °C). (f) The cross-sections of the RIS along the principle axes R_{MAX} and R_{MIN} of Listvianski anthracite thermally treated at various temperatures (400, 600, 800 and 1000 °C).

distinctly higher than the anisotropy of the texture of lower rank anthracites (Moszczenica and Walbrzych) (Fig. 5).

From the point of view of textural transformation during thermal treatment up to 1000 °C, we can

distinguish three groups of anthracites: (i) Listvianski; (ii) Svierdlovsk, La Mure and Donetsk; (iii) Moszczenica and Walbrzych. It seems that the character of alterations of individual anthracites is the result of the degree of their original metamorphism and, in a

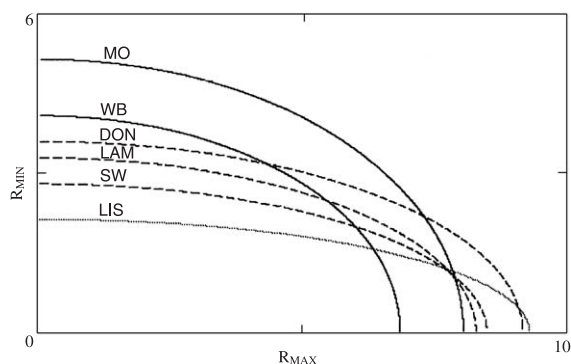


Fig. 5. The cross-sections of the reflectance indicating surfaces along the principle axes R_{MAX} and R_{MIN} of anthracites studied thermally treated at the temperature of 1000 °C. WB—Walbrzych, MO—Moszczenica, DON—Donetsk, LAM—La Mure, SW—Svierdlovsk, LIS—Listvianski.

consequence, the chemical structure and the texture of initial samples.

4. Conclusions

1. R_{max} and R_{min} values increase together with the temperature of treatment for anthracites studied, except for Listvianski sample. The higher the rank of the initial sample, the lower the relative increase of reflectance values but the greater the final reflectance values obtained at 1000 °C.
2. Donetsk, La Mure and Svierdlovsk anthracites reveal insignificant changes of reflectance values below 600 °C, while in the case of Moszczenica and Walbrzych anthracites important changes of reflectance values already occurred at about 400 °C. This suggests that above meta-anthracites underwent higher degree of metamorphism than Moszczenica and Walbrzych anthracites, corresponding to about 200 °C thermal treatment in the laboratory. However, additional metamorphism factors such as pressure and/or shears should be taken into consideration.
3. The changes of R_{ev} values during heating indicate that the alterations of the chemical structure of BSU are stronger for the lower metamorphosed anthracites than for the higher ones.
4. The anisotropy of the texture of meta-anthracites is distinctly higher than the anisotropy of the texture

of lower rank anthracites after heating at 1000 °C. There is a clear relation between the degree of anisotropy of the final texture and the rank of the parent anthracites.

5. It was stated that under thermal treatment up to 1000 °C, the lower metamorphosed anthracites are more supplied for alteration of chemical structure of their BSU, while the higher rank anthracites are more flexible for transformation of their texture.
6. Even at 1000 °C, particular anthracites retain individuality related to their original structure and texture being the effect of their natural metamorphism.

Acknowledgements

These investigations are supported by the State Committee for Scientific Research (KBN), Poland, as the grant project no. 6 P04 D 074 17.

The authors would like to thank Drs. David Houseknecht and Mark Pawlewicz for the many improvements to the manuscript.

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