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PIOTR PEŁKA*, GRZEGORZ GOLAŃSKI**, PAWEŁ WIECZOREK**

EVOLUTION OF THE STRUCTURE AND MECHANICAL STRENGTH OF A COAL PARTICLE DURING COMBUSTION IN THE ATMOSPHERE OF AIR AND THE MIXTURE OF OXYGEN AND CARBON DIOXIDE

EWOLUCJA STRUKTURY ORAZ WYTRZYMAŁOŚCI MECHANICZNEJ ZIARNA WĘGLA PODCZAS SPALANIA W ATMOSFERZE POWIETRZA ORAZ MIESZANINIE TLENU I DWUTLENKU WĘGLA

The research was conducted on the basis of four different types of hard coal and one type of brown coal. There are typical coals commonly used as fuel in Polish CFB boilers. The combustion process was conducted at a temperature of 850°C and the atmosphere of ambient air as well as in the mixture of oxygen and carbon dioxide in different proportions. The research was carried out using specially prepared cubical coal particles with measurements of 15×15mm and also 10×10 mm. The change of the mechanical properties was analyzed based on three parameters, i.e. compression strength, Vickers hardness and fracture toughness. The analysis was supplemented by microscopic images of the surface of the particles using an atomic force microscope. The results obtained clearly indicated the mechanical changes of the coal during its combustion, particularly at the moment of ignition of the char. Moreover, the results correlate very well with the processes of coal comminution that have been described by other authors (Basu, 1999; Chirone et al., 1991) during combustion in the circulating fluidized bed and also explain the sudden change of susceptibility to erosion in the conditions with and without combustion. The measured values can be used as strength parameters in the modelling of the mass loss of coal particles in conditions of circulating fluidized bed combustor that are hard to describe.

Keywords: combustion, comminution, mechanical properties

Badania przeprowadzono na podstawie czterech węgli kamiennych różnego typu oraz jednego węgla brunatnego. Są to typowe węgle energetyczne wykorzystywane powszechnie jako paliwo w kotłach fluidyzacyjnych w Polsce. Proces spalania był prowadzony w temperaturze 850°C w atmosferze powietrza atmosferycznego oraz w atmosferze mieszaniny tlenu oraz dwutlenku węgla w różnych proporcjach. Badania przeprowadzono na spreparowanych do tego celu sześciennych próbkach węgla o wymiarach 15×15 mm oraz 10×10 mm. Zmianę własności mechanicznych przeanalizowano w oparciu

* CZESTOCHOWA UNIVERSITY OF TECHNOLOGY, DEPARTMENT OF THERMAL MACHINERY, ARMII KRAJOWEJ 21, 42-201 CZESTOCHOWA, POLAND. E-mail: pelka@kkt.pcz.czest.pl

** CZESTOCHOWA UNIVERSITY OF TECHNOLOGY, INSTITUTE OF MATERIALS ENGINEERING, ARMII KRAJOWEJ 19, 42-200 CZESTOCHOWA, POLAND

o trzy parametry: wytrzymałość na ściskanie, twardości Vickersa oraz współczynnik kruchości. Analizę uzupełniono zdjęciami mikroskopowymi powierzchni ziaren wykonanymi za pomocą mikroskopu sił atomowych. Otrzymane rezultaty wskazują na bardzo wyraźne zmiany wytrzymałościowe węgla podczas jego spalania, szczególnie w chwili zapłonu karbonizatu. Uzyskane wyniki bardzo dobrze korelują z opisywanymi przez innych autorów procesami rozdrabniania węgla (Basu, 1999; Chirone et al., 1991) podczas spalania w warunkach cyrkulacyjnej warstwy fluidalnej. Tłumaczą gwałtowną zmianę podatności na erozję w warunkach bez spalania oraz z towarzyszącym spalaniem. Rezultaty badań mogą posłużyć jako parametry wytrzymałościowe w modelowaniu ubytku masy ziaren węgla w trudnych do opisania warunkach cyrkulacyjnej warstwy fluidalnej.

Słowa kluczowe: spalanie, rozdrabnianie, właściwości mechaniczne

1. Introduction

In this paper, analysis was carried out on the chosen strength parameters of coal, i.e. compression strength, Vickers hardness and fracture toughness during individual combustion phases. The real values of the parameters facilitate the understanding of the mechanisms of mass loss as a result of comminution during individual combustion phases and consequently their prediction (Pelka, 2011). Comminution processes, i.e. fragmentation and erosion, play a significant role in the process of combustion of solid fuel, especially in the conditions of CFB. They have been the subject matter of research for many scientific centres (Massimilla & Salatino, 1987; Ray et al., 1987; Salatino & Massimilla, 1985; Gajewski & Kosowska-Golchowska, 2007; Chirone et al., 1989; Kijo-Kleczkowska, 2010, 2012). Undoubtedly their meaning in the combustion process is unusually significant. In particular, the erosion process which changes the mechanism of coal combustion during all the individual phases of combustion, but first of all during the process of char combustion (Pełka, 2009). This generates additional mass loss from the mother particle. As a result of contact between the fluidized bed material and the burning particle from its surface the natural barrier is removed from the incombustible part of coal. As a consequence, the diffusion of the oxidant to the reaction surface and also the exhaust gases to the outside is facilitated. In the case of combustion in the fluidized bed this is especially important for the sake of greater gas velocity in the combustion chamber. Fine particles are ripped off next to the ash from the mother particle. According to Basu (Basu & Fraser, 1991) they are less than 100 mm. Additionally, the process of percolation (Chirone et al., 1991) which proceeds during the process of burning out constantly weakens the particle structure which intensifies the erosion mass loss. The burning particle in time gets through to the upper zone of combustion chamber. If in this zone the weakened coal particle has contact with the material of the fluidized bed, then the finest particles formed do not have the possibility of burning out before they are elutriated to the separator of solid material. We know that the probability of separation decreases with the size of the particle. Hence, the smallest particles that are not separated by means of the separator constitute the lost incomplete combustion. This loss can even exceed 4% (Hupa, 2008; Ghadiri & Zhang, 2002). For this reason, fly -ash from the electrofilter is recirculated to the combustion chamber in order to burn the coal content. The model of mass loss of burning coal particles in conditions similar to the upper zone of combustion chamber was presented by Pelka (2011). The author in question used the relationship of the mechanical mass loss of brittle material proposed by Zang, Ghadiri (2002) in the equation of the overall mass loss of the coal particle. However, according to the aforesaid author the complete verification of the model requires the real values of Vickers hardness and fracture toughness. The particle undergoes many physical and chemical processes during the combustion process which weaken the surface and internal structure. It has a direct affect on the intensity of mechanical comminution as a result of erosion.

Getting acquainted with these parameters in the subsequent combustion phases is the basis of the research described in this work. For the sake of common attempts of coal combustion in the atmosphere that is enriched with oxygen this part of the research was carried out on the mixture of O_2/CO_2 in the following proportions: 21/79; 30/70; 40/60.

Additionally, in order to understand the processes during the combustion process better, this analysis has been supplemented by the images of the particle surface from the atomic force microscope.

2. Test stand and methodology

In the research conducted, an analysis of the change of the mechanical properties of hard coal types from the following Polish coal mines have been carried out: Piast, Sobieski, Staszic, Murcki and brown coal from Belchatow mine. The results of the proximate analysis of all the tested coals have been presented in Table 1.

TABLE 1

The result of proximate analysis of the tested coal types

Coal type	Mine	Technical analysis				
		Volatile matter *V*, %	**Moisture *W*, %**	**Ash *A*, %**	**Fixed carbon *FC*, %**	**Lower heating value *Q*, kJ/kg**
		%	%	%	%	kJ/kg
hard	Sobieski	28.91	18.98	9.98	49.94	23488
hard	Piast	31.34	8.26	8.52	51.88	25659
hard	Staszic	30.09	2.66	2.36	64.08	31198
hard	Murcki	30.9	4.3	8.2	63.38	30110
brown	Belchatow	37.11	14.46	18.42	30.01	16165

The combustion process was carried out using the test stand whose scheme is shown in Fig. 1. The coal particles were inserted into the combustion chamber by means of a mobile system where the tested particle was placed on the handle of an extensometer branch scale. The combustor chamber was equipped with a sight glass made from quartz glass which enabled the observation of the combustion process during its entire course.

As the aim of the work was the determination of the chosen strength parameters of coal in all phases of combustion, it was therefore necessary to break the combustion process down into all its stages. The particles with relation to the strength tests after drying, i.e. after the first stage of combustion were according to the Polish norm (PN-80/G-04511) in a separate heating chamber. The other coal particles during the next three phases were as a result of breaking up the process on the basis of the mass loss observed on the branch scale, as well as the observation of the process through the means of the sight glass. The strength tests in the range of three chosen parameters in all the phases were carried out using a series of 10 particles for each parameter separately. The final result was obtained by means of averaging out the individual results.

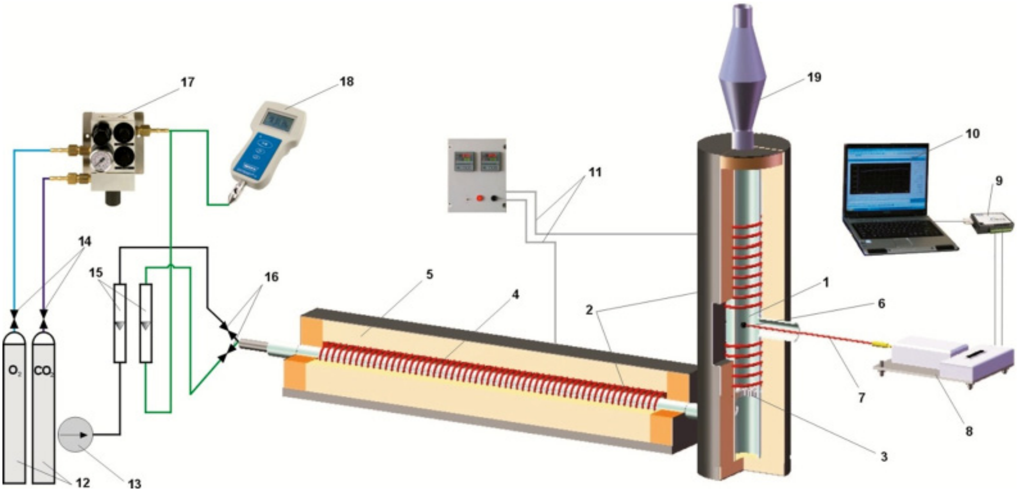


Fig. 1. Schematic diagram of the test apparatus: 1 – combustion chamber, 2 – heating elements, 3 – distributor, 4 – preheater, 5 – thermal insulation, 6 – coal particle, 7 – S–type thermocouples, 8 – laboratory scale, 9 – measuring card, 10 – computer, 11 – system of regulating and measuring temperature, 12 – gas cylinders, 13 – air compressor, 14 – pressure regulators, 15 – rotameters, 16 – flow regulating valves, 17– gas mixer, 18 – oxygen analyser, 19 – ventilation duct

The tests on the coal samples encompassed the following:

- static compression strength, (determination of the maximum force),
- Vickers hardness,
- fracture toughness by means of the stress intensity rate.

Static compression strength and fracture toughness were realized for cubic coal particles with measurements of 15×15 mm. These tests were carried out by means of a servo-hydraulic testing machine MTS810 type. The velocity of compression was 0.02 mm/s. Moreover, in the case of raw coal, tests were run parallel and perpendicular to the coal lamination. However, the measurement of hardness by means of the hardness testing machine- Future Tech FV-700, was carried out to comply with penetrator loading 10 kG (98.07 N). In the case of coal after burning out, the volatile matter due to the high level of brittleness required a lower penetrator loading of kG, 1 kG and also 0.05 kG to be used for the measurement of hardness. This may generate greater errors during the readings of traces and consequently the values of hardness.

The fracture toughness K_C was carried out by means of the same servo-hydraulic testing machine with the same velocity of compression. The tests were realized by using the methodology proposed by Klepaczko (Klepaczko et al., 1984) for raw coal types rather than after drying. The tests during the subsequent phases of combustion were not conducted as a result of the excessive weakness softening of the particle structure.

3. Results

3.1. Static compression strength

The first stage of the research was to conduct a static compression of all coal types and determine the maximum destructive force. In Table 2, the results obtained in the next combustion phases have been presented. For the individual coal types, different values of maximum destructive force, as well as the rather substantial standard deviation were registered. As anticipated, the decisively lowest value of compression strength for brown coal was measured – 1100.3 N. This is also contributed by the fact that brown coal is younger than hard coal, thus containing the greatest volatile matter (37.11%) and also the greatest content of ash (18.42%). The hard coal types are characterized as having several times greater value of force from 6751.6 N – in the case of the Murcki mine to 3576.8 N – in the case of the Piast mine. The analysis obtained in the subsequent combustion phase clearly points towards the significant changes of strength that occur during the next combustion process. As a result of drying, a decreasing maximum of force of was noticed in the case of coal from, Staszic, Sobieski and Murcki even 50%, whereas in the cases of the other types of coal, the value of force remain at the same level when taking account of the standard deviation value. This aspect would indicate that the drying process may have an impact on the change of strength of coal, thus weakening it in different areas. It seems that this range depends more on coal morphology than on its moisture. The coal types indicating the greatest weakening (Murcki, Staszic) are characterized by extreme moisture content. In moving on to the subsequent combustion phase, i.e. the completion of combustion of volatile matter and the ignition of a char, we can note the complete change in the value of the maximum strength value for all the tested coal types. The difference for coal from Sobieski equals 3065.11 N, which indicates a weakening of the coal structure with reference to raw coal by over 25 times. During the next combustion phase of the char, i.e. after 60s the maximum strength force stabilizes at a similar level, especially in the case of coal from Piast and Sobieski. A different feature is indicated by brown coal for which the value of the strength force decreases and reaches a value of 34 ±3.5 N only after 10s from the completion of the combustion of volatile matter. Aside from the difference of the morphological structure and the chemical composition, the other cause is the intensity of the combustion process. The time of combustion of brown coal particle is about four times shorter as a result of the low value of fixed carbon and the high value of volatile matter. Fig. 2 presents the hypothetical mass loss of tested coal particles in the mixture O_2/CO_2 at a gas concentration of 30%/70%. The mass loss in the atmospheric air differs only in terms of the somewhat longer time of overall mass loss. Only 30% of fixed carbon remains after the process of burning out of volatile matter which ignites earlier during the combustion of volatile matter and burns faster with relation to hard coal types. It is necessary to state that actually for this reason a drastic weakening of the structure occurs over a short time.

Due to the excessive change in the particle geometry after the combustion of volatile matter, the measurement of strength force for coal from Staszic mine was not possible.

TABLE 2

Result of compression of coal types in the different phases of combustion

Coal phase / Coal type	RAW	After drying	After volatile matter combustion	Ahar (after 60s)
	Mean value *F* [N] / standard deviation [N]			
Murcki	6751.6/2862.7	2542.8/1851	268.2/227	734/52.5
Staszic	4361/1110.2	3433.4/1355	798.4/462.3	-
Belchatow	1100.3/627.9	1168/297	421.7/247.9	34/3.5 (after 10s)
Sobieski	5496/1256.6	3195/71.3	158.5/101.5	205.5/61.5
Piast	3576.8/329.1	3663.4/914	424.7/55.4	497/15

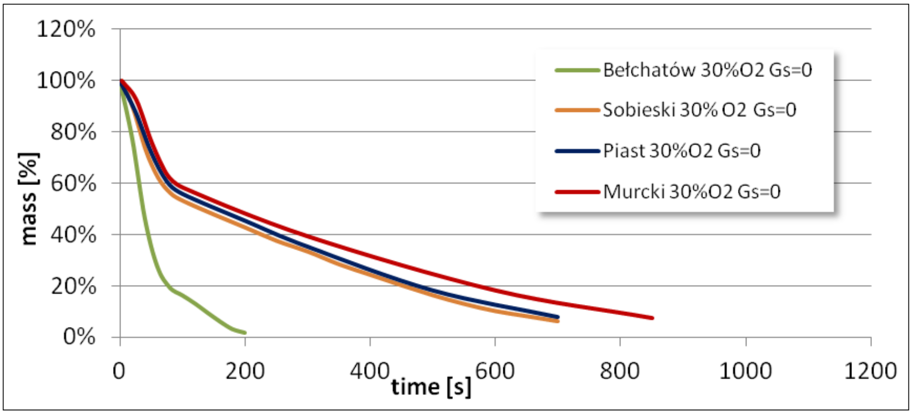


Fig. 2. Mass loss of coal particles burned in the atmosphere of mixture oxygen 30% and carbon dioxide 70% at a temperature of 850°C

The next stage of the research was the estimation of the differences of levels and rates of weakness of coal structure in the different atmospheres of combustion, i.e. air and mixture of oxygen and carbon dioxide. The tests in the air were carried out for coal types from Sobieski, Murcki, Piast and Belchatow, however, in terms of the mixture of oxygen and carbon dioxide this was done with relation to coal from Sobieski coal only. Sobieski coal has the best properties of preserving geometry during particle combustion, moreover, it does not undergo fragmentation and swelling. In order to analyze the change of structure weakness during the char combustion the range of the test was expanded. The results presented in Table 3 and Table 4 indicate that during the course of the process of char combustion further weakness of the structure does not occur.

Maximum strength forces do not change essentially after burning out of the volatile matter with the ignition of the char. By taking the standard deviation values for individual times into account, we can state that they oscillate around similar values. It has also been stated that the change of combustion atmosphere in the tested range does not affect the weakness of coal structure, but merely shortens the time period of particle burning. Hence, the number of measurements of strength force decreases with the increase in the amount of oxygen in the mixture.

TABLE 3

Results of compression of coal from Sobieski mine in the air and different mixtures of O_2/CO_2

Coal phase	Mean force. *F* [N] / Standard deviation [N]	Coal phase	Mean force. *F* [N] / Standard deviation [N]
atmospheric air		char combustion 30s	356.5/82.5
raw	5496.0/1256.6	char combustion 45s	228.5/61.5
following drying	3195.3/112.7	char combustion 60s	176.33/60.45
following volatile matter combustion	122.8/101.5	char combustion 120s	177/23.8
char combustion 15s	256.3/104.2	mixture O_2/CO_2: 30%/70%	
char combustion 30s	162/93	following volatile matter combustion	236.5/124.5
char combustion 60s	198.6/170.1	char combustion 15s	54/12
char combustion 90s	68.7/3.1	char combustion 30s	307/69.07
char combustion 120s	277/176	char combustion 60s	275.5/85.5
char combustion 150s	106/27.1	mixture O_2/CO_2: 40%/60%	
mixture O_2/CO_2: 21%/79%		following volatile matter combustion	158/105
following volatile matter combustion	312.5/30.2	char combustion 15s	34.5/17.5
char combustion 5s	256.7/68.7	char combustion 30s	155.5/9.5
char combustion 10s	391.7/74.2	char combustion 60s	65
char combustion 15s	359.3/60.3		
char combustion 25s	273/10		

TABLE 4

Results of compression of coal from Murcki, Piast and Belchatow mines following combustion of volatile matter and during the combustion of char

Coal	Combustion phase	Mean force, *F* [N] /Standard deviation [N]
Murcki	following volatile matter combustion	268.2/227
	char combustion 15s	724
	char combustion 30s	270.5
	char combustion 60s	734/52.46
Piast	following volatile matter combustion	424.67/55.37
	char combustion 15s	531/64.44
	char combustion 30s	459.67/285.66
	char combustion 60s	497/14
	char combustion 90s	308.33/81.29
	char combustion 120s	265.5/29.5
Belchatow	following volatile matter combustion	421.67/247.94
	char combustion 1s	54.5/3.5
	char combustion 5s	23/4.58
	char combustion 10s	34/3.5

For the sake of high values of standard deviation, which in some cases even reached 50% it was decided to measure the maximum force acting parallel and perpendicular to the lamination of coal in the case of the coal type from Sobieski mine. The results have been presented in Table 5.

On the basis of these values, we can state that the mechanical properties are anisotropic and strongly depend on the direction of lamination. The difference of compression force can equal even 100%. Moreover, it may be noticed that the denoted standard deviation for the perpendicular direction is also high which may result from the difficulty of determining the direction of the lamination and also the changing lamination inside the particle.

TABLE 5

Results of compression of coal particles perpendicular to and parallel with coal limitation

Direction of coal compression	**Maximum force *F*, [N]**	**Mean force, *F* [N] /Standard deviation [N]**
perpendicular to	7921	5736.50/2239.50
parallel with	3609	3397.33/268.73

3.2. Measurement of hardness

The second parameter determined within the confines of the realized work was Vickers hardness *HV.* This parameter according to Ghadiri i Zhang (2002) is equal to the fracture toughness K_c in terms of the essential parameter in describing the susceptibility of brittle material to comminution as a result of the collision of particles. The results of Vickers hardness obtained, (Table 6) confirm the principle that the hardness of coal increases with the amount of fixed carbon. Thus, the greatest hardness was characterized by coal from Staszic mine (FC = 64.08), but the lowest (FC = 30.01), was characterized by brown coal from Belchatow. The greatest change of hardness was similarly observed in the case of the test of static compression following combustion of volatile matter. The drying process weakens the hardness of coal, but only to an insignificant extent. The mean results obtained for hard and brown coal types are at the same level with relation to the values of raw coal, but the standard deviation this time indicates a good level of consistency in terms of results. All the tests were carried out perpendicular to the lamination. The measurement of char hardness during the process of combustion was only possible for coal from Sobieski mine, but only after a time period of combustion of 5, 10, 15 and also 25s for the sake of degradation of the coal surface structure.

TABLE 6

Vickers hardness for coal types in different combustion phases

Coal phase / Coal type	**RAW**	**After drying**	**After volatile matter combustion**	**Char**
	HV10×10^9 [N/m^2] mean value/standard deviation			
Sobieski	0.1864/ 0.021	0.1684/0.094	0.0618	0.055
Bełchatów	0.0654/ 0.0056	0.0875/0.0158	-	-
Piast	0.1962/ 0.0179	0.276/0.034-	-	-
Staszic	0.3414/ 0.0525	0.298/0.07-	-	-
Murcki	0.2191/ 0.0316	0.32/0.01-	-	-

3.3. Fracture toughness K_C

The fracture toughness rate according to the Klepaczko method (Klepaczko et al., 1984) was determined and the tested particle was characterized according to Fig. 3 geometry. The particles prepared for testing are presented in Fig. 4.

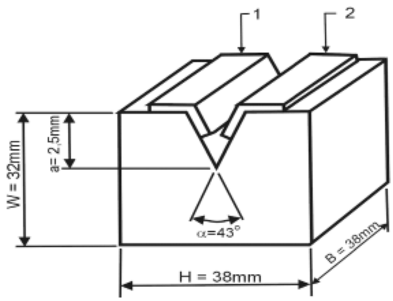


Fig. 3. Geometry of coal particles prepared for the measurement of fracture toughness

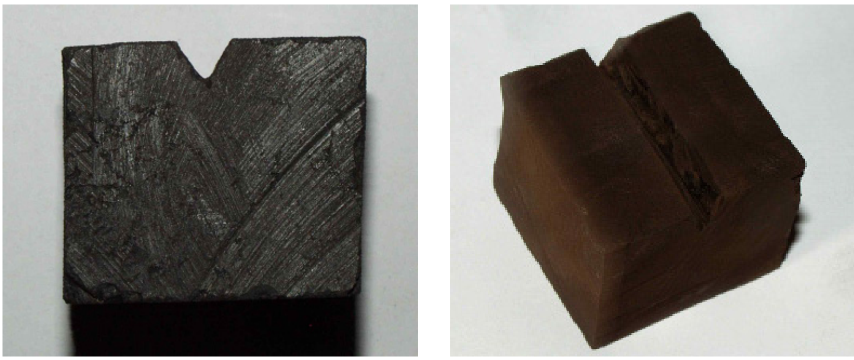


Fig. 4. Coal particles from Sobieski and Belchatow mines prepared for the measurement of fracture toughness

The values of the fracture toughness rate for raw coal from the Sobieski mine after drying, as well as following the volatile matter burning out are presented in Table 7. Due to the excessively large changes in the particle structure and its fragmentation, the determination of the fracture toughness rate during char combustion has not been characterized. It should be noted that the particles used in this section of the test were large in size according to the Klepaczko procedure, i.e.: 38/38/32. In the case of such large quantities of particles the time of the combustion process of the volatile matter is long and amounts to 400 seconds. For this reason, the char combustion phase begins long before the completion of the process of combusting the volatile matter as indicated in Fig. 5. It may be recognized that the obtained values of fracture toughness after the volatile matter burning out have very similar values to the real value of the fracture toughness rate in the phase of char combustion.

Fig. 5. Combustion of volatile matter and char particles during the tests on fracture toughness rate

The results obtained suggest that all the types of coals particles tested without moisture possess similar values of fracture toughness to the value in the case of raw coal only. In the case of coal from Sobieski mine, this value clearly increased which indicates the decrease of brittleness in the analyzed stage. It suggests that without running the combustion process the mass loss of the mother particle as a result of collision according to the equation proposed by Pelka (2011) would not be so high, similar to or even less than in the case of raw coal. In order for the equation to be fulfilled in the case of comminution which takes place together with the combustion char process, the value of the fracture toughness rate should significantly decrease its value. On the basis of the obtained values, it may be acknowledged that this is in reality the case. The mean value of the fracture toughness rate after combustion of the volatile matter is one order of magnitude smaller than $K_c = 0.05381 \times 10^6$ Nm$^{-3/2}$ with relation to its value for coal without moisture $K_c = 0.4490 \times 10^6$ Nm$^{-3/2}$. The changes in the parameters of the mechanical strength previously observed suggest that we may expect only a further insignificant decrease in the value of the fracture toughness rate, which in turn will lead to the decrease in the level of brittleness and consequently, the increase in its mass loss in the erosion process in conditions of the fluidized bed.

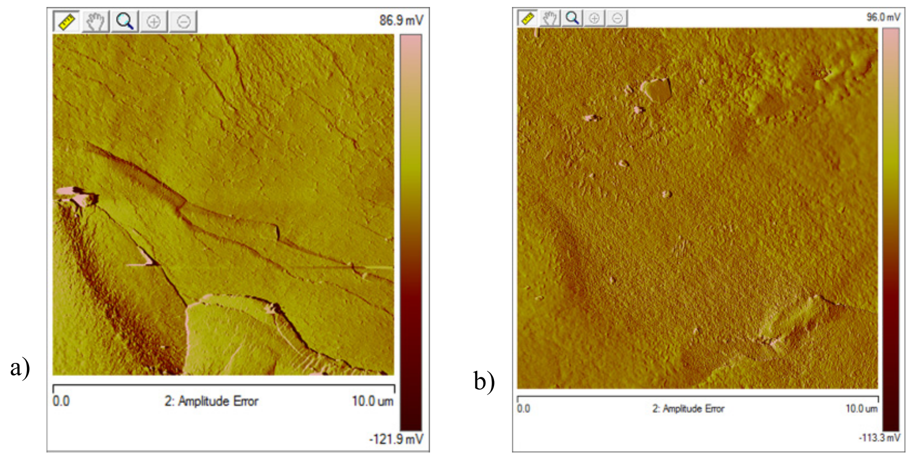
TABLE 7

Values of fracture toughness K_C for coal types in different phases of combustion

Coal phase / Coal type	**RAW**	**Following drying**	**Following volatile matter combustion**
	$K_c \times 10^6$ [Nm$^{-3/2}$]		
Murcki	0.175±0.0216	-	-
Sobieski	0.1891 ±0.083	0.4490 ± 0.2005	0.05381±0.04517
Piast	0.6127 ± 0.1241	0.5828 ± 0.0370	-
Staszic	0.3954 ± 0.1904	0.3466 ± 0.3348	-
Belchatow	0.1339 ±0.0788	0.0816 ± 0.0033	-

4. Analysis of particle surface structure by means of atomic force microscope

The application of the multimode microscope in the tests enables the presentation of the real surface structure in a micro- and nano-metrical scale. A semi-contact mode was used in order to determine the characteristic parameters directly from a sample and not from a picture as in scanning microscopy. The limitation of this method is scanning of an area that is often the size of a few square micrometers. The analysis showed in most cases smooth surfaces that arose as a result of fractures along a cleavage plane as illustrated in Fig. 6.



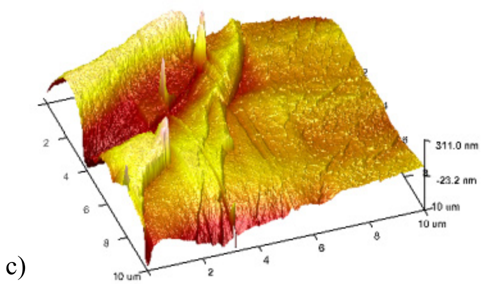


Fig. 6. Topography of surface of raw coal from Sobieski mine: a), b) image 2D, c) image 3D, scanning range 10×10 mm

The state of development of the surface area was insignificant and amounted to about 2%. On the surface beside the fine particles shown in Fig. 7a, structures of much greater dimensions occurred, as shown in Fig. 7b. In the case of drying coal, the increased surface area difference rose to 8-25%. The character of the fracture (along the cleavage plane) was maintained as indicated in Fig. 8a. As in the case of raw coal greater grains occurred in the structure with similar hardness as shown in Fig. 8b.The parameters of the surface roughness for similar scanning surfaces increased in terms of *Ra* from 17 to 190 nm. The other parameters also increased as illustrated in Table 8: *Ra*, *R*max, *Rq*. (*Ra* – aritmetic average of profile deviation, *Rq* – average mean square deviation, Rmax- maximum vertical distance between the highest and the lowest surface point).

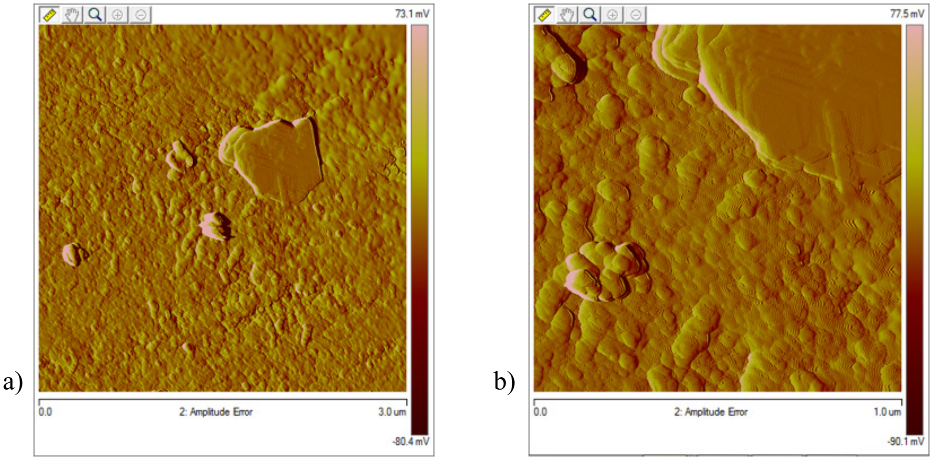


Fig. 7. Raw coal: a) topography 3×3 mm, b) topography – 1×1 mm

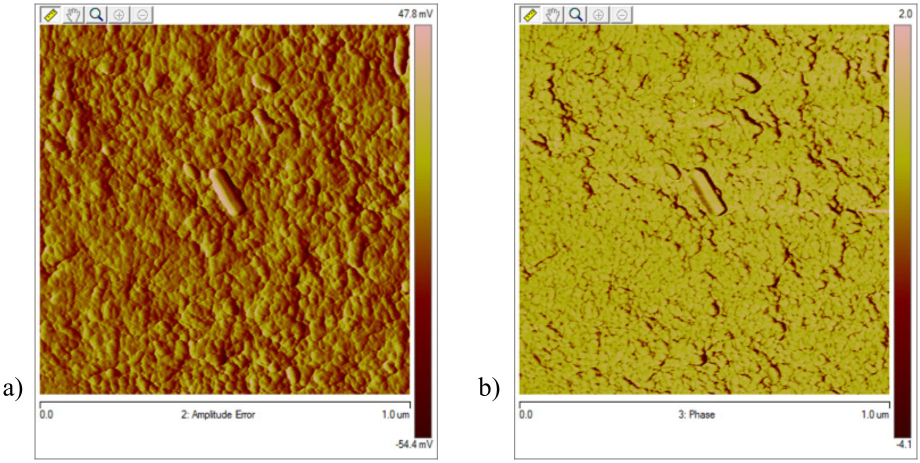
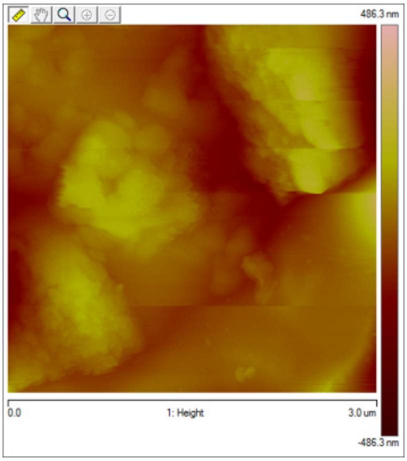


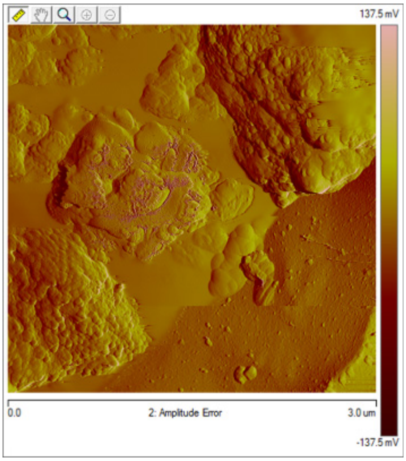
Fig. 8. Drying coal: a) topography 3×3 mm, b) contrast of mechanical properties – 1×1 mm

TABLE 8

Parameters of surface roughness for a – raw coal, b – drying coal

a) Scanning area 108 mm^2			
SAD [%]	*Ra* [nm]	*R*max [nm]	*Rq* [nm]
8.04	25.6	664	47.2
b) Scanning area 80 mm^2			
25.9	190	1554	240

a) 

b) 

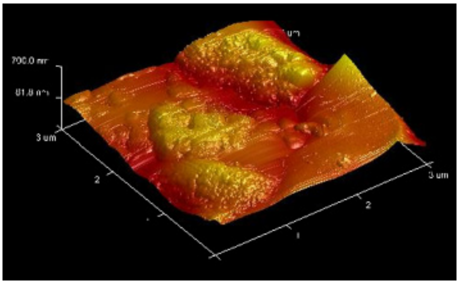
c) 

Fig. 9. Following combustion 15 s: a), b) image 2D, c) 3×3 mm – topography, 3D

The granular structure was maintained, but the grains are clearly more spherical than in the case of the previous samples. There is a change of character of the substance obtained after combustion of volatile matter in terms of the fracture from a cleavable to a granular one. The sample possesses a much more developed surface structure. The parameters of the surface roughness obtained for the same sample cannot be compared with the precious ones, because the scanning area was 10 times smaller which leads to a lower average value. In the sample there are clearly visible changes to the granularity as large particles appear next to the fine grains. The analyzed preparations are characterized by a very similar average diameter of granularity. In terms of combustion of the char in a timescale of 15 seconds a distinctive division into the areas takes place, whereby the effects of burning occur as well as areas maintaining the initial structure as displayed in Fig. 9. The roughness of the areas that are not over-burnt is similar to the raw probes. In the over-burnt areas the rate of development increases to about 56%. The parameters and profiles of both surfaces are presented in Table 9 and Fig. 10. The left side of the profile presents the burning area, however the primary area has been indicated on the right side. The areas beside the topography also differ in terms of hardness – the surface that is not over-burnt is harder with comparison to the over-burnt surface, as well as the average diameter of the over-burnt grains are approximately three times bigger than the ones not over-burnt.

TABLE 9

Parameters of surface roughness after combustion lasting 15 s

Surface area	3.11 μm^2	2.41 μm^2
SAD	56.9%	12.8%
Rq	123 nm	41.4 nm
Ra	105 nm	26.1 nm
Rmax	583 nm	447 nm

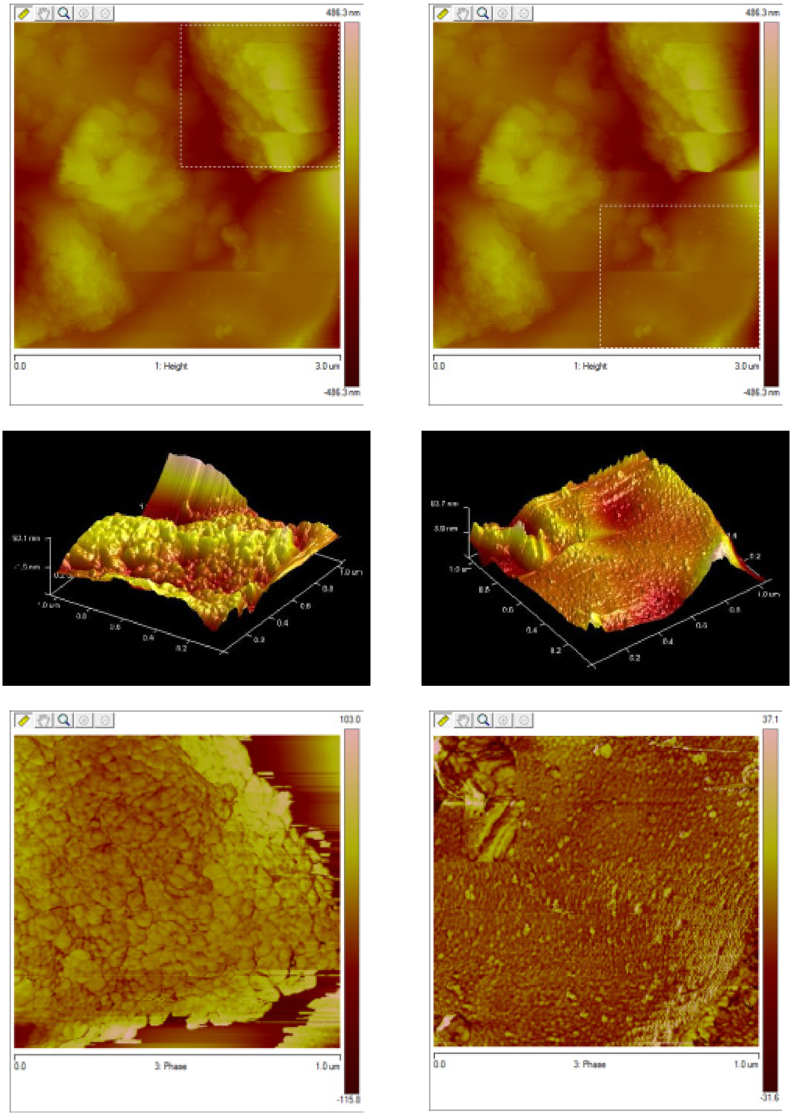


Fig. 10. Topography of surface and roughness after combustion lasting 15 s

The surface of the sample burnt for 30s undergoes further development as shown in Fig. 11. There is an increase in both the amount of big grains as well as in terms of their diameter. A further increase in the length of time of burning to 60s does not cause an increase in surface development. It stays at a level of about 40%, but this result is somewhat lowered owing to the smaller area taken for analysis. On the small surface it is possible to find sporadic areas that do not burn as illustrated in Fig. 12 b-d. For the probes that have undergone the longest length of time in terms of burning, the process of progressive burning out is visible, while there is also a lack of areas with a primary structure that do not burn. The grains in the burning areas appear in two morphologies: soft grains with diameters of about 500 nm and areas with a granularity lower than 70 nm.

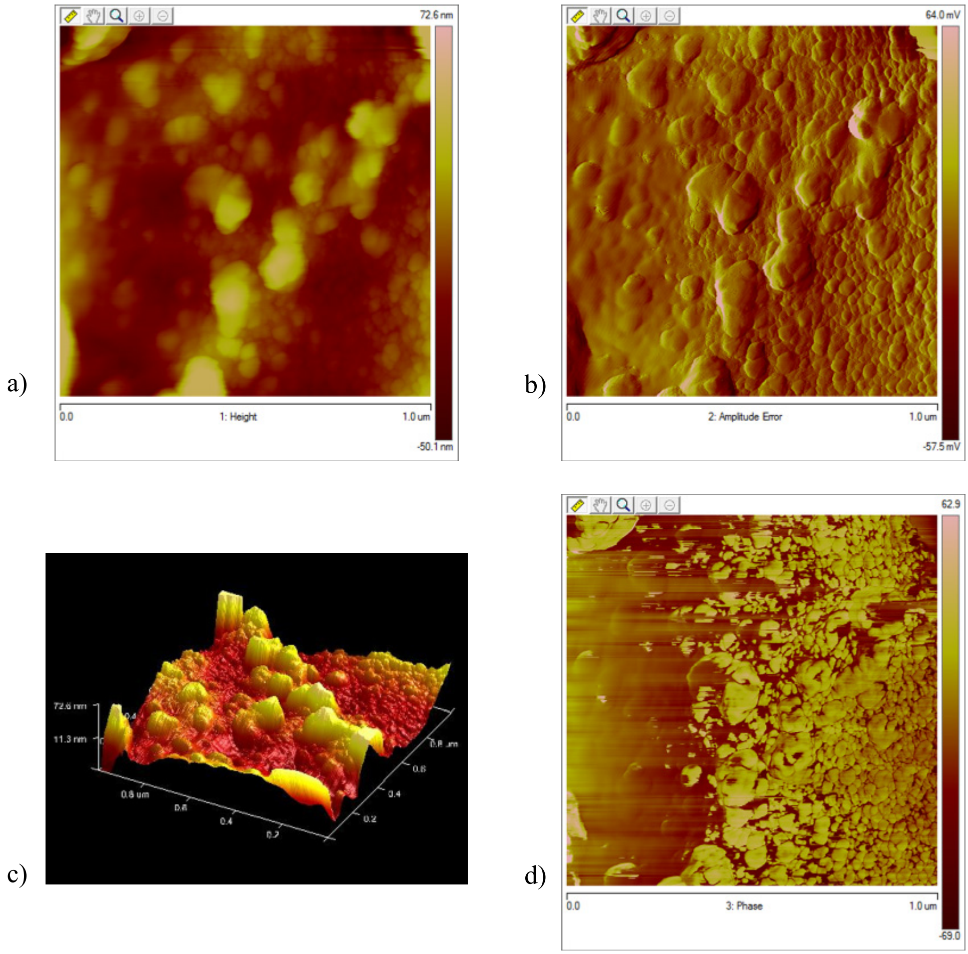


Fig. 11. After combustion lasting 60 s: a, b) topography, c) image 3D, d) phase contrast

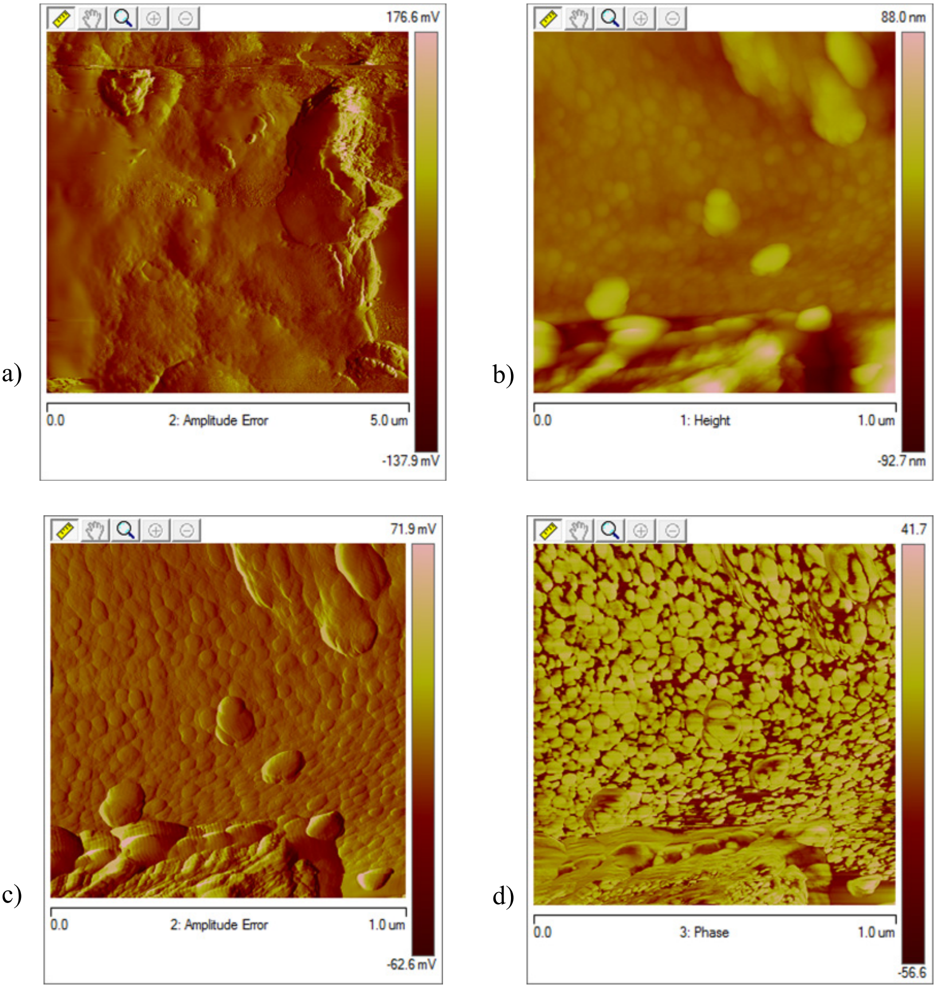


Fig. 12. After combustion lasting 60 s: a) Canning plane 5×5 mm, b, c) 1×1 mm – topography, d) phase contrast

4. Conclusions

The research has been conducted by using Polish hard coal and brown coal. All coal types in a raw state are characterized by decisively varied strength properties. The difference of static compression strength obtained for hard coals was 100%, while 600% for brown coal. A similar difference was observed during the test of fracture toughness, whereas a somewhat lower one for Vickers hardness. It points to a very wide range of mechanical properties for coal types and the difficulty in the accuracy of their estimation. Nevertheless, from the point of view of comminution, the more important aspects are the parameters and their change during the combustion process. In all cases, the results obtained indicate change in all the strength parameters. This change is strictly related to running one combustion phase after another which consist of different physical and chemical processes.

The least influential change on the strength parameters of coal is that of the drying process. In almost all cases a small change of values was observed and by taking account of the quite high standard deviation value, we can state that the drying process does not significantly change the strength of a coal particle. The next combustion phase is devolatilization and combustion of volatile matter. It is obvious that these processes overlap and are in fact inseparable, thus, in the research carried out on the changes, the strength of coals after combustion of volatile matter was determined. The results obtained indicate a drastic decrease in all the tested parameters – one order of magnitude for all the hard coal types. In the case of brown coal, the difference is not so high which results from the age of coal and its chemical composition, but it is quite distinctive. It should be noticed that at the moment of the completion of combustion of volatile matter the ignition of char surface is observed. Hence, the key phenomenon causing the weakness of the coal structure seems to be the process of internal and first and foremost, external char combustion. Further char combustion does not weaken the coal structure in principle, and in some cases it even makes it somewhat more rigid.

The test methodology accepted for the practice of determining the strength parameters is correct, although it did not manage to directly determine all the values of the accepted scope for char combustion. The results presented correlate with the processes of coal comminution very well as described by other authors during the process of combustion in conditions of a circulating fluidized bed and explain the sudden change of susceptibility to the erosion process for pure mechanical attrition with and without assisted combustion. They can be used as parameters rendering the modelling of the mass loss of coal particle possible in conditions of a circulating fluidized bed that are difficult to describe.

The change of atmosphere from air to mixture of O_2/CO_2 and increase of the oxygen in the mixture do not affect changes in the mechanical properties of the tested coals. Only the subsequent rapidly progressing phases of combustion were observed and their associated faster coal particle mass loss.

The analysis of the evolution of the structure by means of atomic force microscope indicate the causes of change in the mechanical strength. The observed evolution of grains constitute the coal particle structure, i.e. the increase of the average size of grains, increase in roughness, difference of hardness on the phase boundary all point to the increase of susceptibility of coal to comminution during the combustion process.

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