

Production of high-temperature and high-resistivity semicoke from coals of “Shubarkol komir” JSC

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ABSTRACT

This study presented the results of an investigation into formation patterns of high-temperature specific electrical resistivity (SER) in coke produced from the coals of JSC “Shubarkol Komir” (Kazakhstan). The study also examined the impact of silica-containing surface modification and the coking temperature regime on the coke's electrical and physico-mechanical properties. This study is relevant because it addresses the need to control the distribution of electrical current and heat generation in the near-electrode zone of ore-thermal furnaces. This can be achieved by optimising the properties of carbon-based reducing agents. Silica modification resulted in a 3.5- to 4.5-fold increase in specific electrical resistivity in the temperature range of 50-300 °C. At 1200°C, the increase was observed to be 1.4- to 1.5-fold. Three distinct temperature regions of resistivity change were identified: 50-400 °C (contact-degassing), 400-800 °C (intensive thermodestruction), and above 800 °C (structural ordering). The increase in SER was attributed to the formation of a dielectric silica film. This was accompanied by increased ash content and reduced bulk density. A compromise between the electrical and mechanical properties of the coke was observed. The results expand understanding of the mechanisms underlying high-temperature conductivity in carbonaceous reducing agents. These findings can be applied for targeted control of the electrical regime in electrothermal processes.

Keywords: high-resistivity coke; coal; coking; specific electrical resistivity; carbonaceous material; reducing agent.

INTRODUCTION

Semicoke is a carbonaceous material produced from low-metamorphic coal by low-temperature carbonization, typically in the range of 600–800 °C. It is characterized by high chemical reactivity, a rapid carbon reaction rate, and relatively low sulfur and phosphorus contents [1,2]. This process differs from conventional metallurgical coke production, which is generally carried out at higher temperatures of approximately 1000–1100 °C [3]. Semicoke production also generates coal gas and coal tar as by-products [4]. Due to its properties and comparatively lower pollutant emissions, semicoke, also known as blue coke, is considered a promising carbonaceous material for metallurgical and electrothermal applications [5,6].

A key advantage of semicoke production is its adaptability to a wide range of coal types, including coals with high volatile matter content and low caking ability. In addition, the process does not necessarily require the complex raw-material preparation typically associated with conventional metallurgical coke production, creating opportunities for large-scale industrial application [7,8].

In electrothermal processes, including the production of ferrosilicon, silicon, titanium, and other ferroalloys, the requirements imposed on carbonaceous reducing agents differ from those traditionally applied to metallurgical coke. In addition to mechanical strength, reactivity, and thermal stability, specific electrical resistivity (SER), particularly at elevated temperatures, is an important functional characteristic of the reducing agent.

The electrical resistivity of carbonaceous reducing agents affects the total electrical resistance of the furnace charge and, consequently, electrode immersion, energy distribution, and the thermal conditions within the reaction zone [9,10]. Therefore, the use of carbonaceous materials with appropriate electrical characteristics is important for maintaining stable furnace operation and efficient utilization of electrical energy [11].

The specific electrical resistivity of carbonaceous materials is influenced by several interconnected factors, including carbon structure, ash composition and mineral matter, degree of graphitization, porosity, and thermal treatment conditions [12]. These factors determine the formation and continuity of electrically conductive pathways within the carbon matrix and therefore control the electrical behavior of the reducing agent.

Among these factors, the degree of graphitization plays a particularly important role in determining electrical resistivity. Increasing structural ordering and graphite content generally promotes the formation of regular sp²-bonded carbon structures, facilitates electron transport, and consequently decreases specific electrical

resistivity [13,14]. Graphitic materials therefore exhibit substantially lower electrical resistivity than less structurally ordered carbonaceous materials [15,16]. Polycrystalline carbon materials generally exhibit higher electrical resistivity than highly ordered graphite because crystal boundaries, structural defects, and pores hinder charge transport. Their electrical properties may also vary depending on the orientation of graphite-like structural units [17]. Consequently, the electrical resistivity of carbonaceous reducing agents depends not only on their chemical composition but also on the structural transformations occurring in the carbon matrix during thermal treatment.

Carbonization temperature is therefore one of the major technological parameters controlling the electrical properties of coke and semicoke. Increasing the treatment temperature promotes the removal of volatile components and heteroatoms and facilitates structural rearrangement and ordering of the carbon matrix. As a result, electrical resistivity generally decreases with increasing carbonization temperature [18,19]. Similar relationships between thermal treatment, structural ordering, and electrical conductivity have been reported for various carbonaceous materials [20]. In addition to the structural state of carbon, mineral matter and surface modification may influence electrical behavior by changing the electrical contact between coke particles and interrupting conductive pathways. This aspect is particularly relevant when silica-containing materials are introduced onto the coke surface. However, the effect of such surface modification on the high-temperature electrical resistivity of metallurgical carbonaceous reducing agents remains insufficiently investigated.

Despite numerous studies on the electrical properties of carbonaceous materials, little information is available on the high-temperature specific electrical resistivity of coke produced from low-metamorphic coals of Central Kazakhstan, particularly coal from JSC “Shubarkol Komir”. Furthermore, the effect of microsilica treatment on the electrical behavior of such coke over a wide temperature range has not been sufficiently investigated.

Therefore, the objective of this study is to determine the effects of coking conditions and microsilica treatment on the high-temperature specific electrical resistivity and mechanical properties of coke produced from JSC “Shubarkol Komir” coal. Particular attention is given to the temperature dependence of SER in the range of 50–1200 °C and to the comparison of untreated and microsilica-treated coke.

Experimental batches of coke were produced using coking regimes developed by the authors and protected by patents of the Eurasian Patent Organization (EAPO). The produced materials were evaluated using standardized methods, including proximate analysis, granulometric analysis, drop and rotary-drum strength tests, and measurement of specific electrical resistivity over a wide temperature range.

We determined the physicochemical and mechanical characteristics of the carbonaceous materials using standard equipment in accordance with relevant GOST procedures.

MATERIAL AND METHODS

Coal from the Shubarkol deposit with an initial particle size of 50-150 mm was used as the raw material for the preparation of the experimental coke batches. Prior to coking, the coal was crushed and sieved, and the 10-25 mm fraction was separated for further experiments.

The selection of the 10-25 mm fraction was intended to provide a relatively uniform feedstock for the semi-industrial coking experiments and to obtain coke of a particle size suitable for subsequent characterization and specific electrical resistivity measurements.

The experimental coke batches were produced in a semi-industrial coal coking unit using high-speed thermo-oxidative pyrolysis (Figure 1). The unit has an internal diameter of 1066 mm in the cylindrical section, a cylindrical-section height of 1400 mm, and an effective volume of 2.3 m³. At an average bulk density of the feedstock of approximately 0.7 t/m³, the unit can accommodate approximately 1.6 t of raw coal.

The apparatus consists of a steel cylindrical body lined with refractory bricks and a truncated conical section at the bottom. The upper part of the unit is equipped with a loading hatch and a nozzle for the discharge of gaseous pyrolysis products, whereas the lower part contains a hatch for unloading the produced coke.

The gaseous products formed during pyrolysis were directed through the discharge nozzle to a combustion device, where the combustible components were burned. After completion of the coking process, the coke was discharged through an inclined vibrating chute and cooled using a water-spray system. The cooled product was subsequently collected for further characterization and testing.

Microsilica, a by-product of high-silicon alloy production, was used to surface-treat the coke. The microsilica consisted predominantly of ultrafine amorphous SiO₂, with a specific surface area of 15-25 m²/g and an SiO₂ content of ≥95%. Owing to its high dispersibility, microsilica can be distributed over the surface of coke particles during treatment.

In the present study, microsilica treatment was investigated as a method for modifying the electrical behavior of coke. Since direct microstructural and phase characterization of the treated coke by SEM/EDS or XRD was not performed, the formation, continuity, and phase composition of a silica-containing surface layer were not assumed as experimentally established. Accordingly, the effect of microsilica treatment was evaluated primarily on the basis of the measured changes in the specific electrical resistivity of the coke.

The experimental program included the production and subsequent characterization of untreated and microsilica-treated coke batches. The physicochemical and mechanical properties of the obtained materials were determined using standardized methods. Particular attention was given to the measurement of specific electrical resistivity over the temperature range of 50–1200 °C in order to evaluate the influence of temperature and microsilica treatment on the electrical behavior of the coke.

PRODUCTION OF HIGH-TEMPERATURE AND HIGH-RESISTIVITY COKE

At the first stage, a batch of high-temperature coke was produced at a coking temperature of 750-850 °C. The process was carried out under oxidative conditions. The combustion temperature was regulated by varying the air flow. It was maintained between 800 and 850 °C.

At the second stage, an experimental batch of coke with increased specific electrical resistivity was produced. This parameter was key to reducing carbonaceous agents in electrothermal processes. The increase in increased specific electrical resistivity was achieved using the patented technology of LLP “PKK Modul”. This technology involved treating the coke with an emulsion based on microsilica, which formed a protective film on the particle surface. This coating reduced their electrical conductivity.

The temperature range of 750-850 °C was selected based on the patterns of thermo-chemical transformation of the carbonaceous material.

In this temperature range, key processes responsible for the formation of the coke structure occurred:

- deep destruction of the organic mass,
- removal of volatile substances,
- formation of the primary carbon matrix,
- development of porosity,
- growth of carbon microcrystallites.

At temperatures below 700 °C, the carbonization processes were incomplete. This increased the content of volatile substances and caused structural instability.

At temperatures above 900 °C, active structural ordering and partial graphitization began. These processes were accompanied by a decrease in electrical resistivity.

Therefore, 750-850 °C is optimal for producing coke with higher specific electrical resistivity and a well-developed porous structure.

The combustion temperature of the coal charge was maintained within 800-850 °C by regulating the blast air flow. This temperature range was selected to ensure:

- stable progression of the pyrolysis process,
- uniformity of the thermal field,
- prevention of local overheating,
- stability in the formation of the coke matrix.

At lower temperatures, incomplete thermodestruction occurred. At temperatures above 900 °C, the graphitization processes intensified, leading to a decrease in specific electrical resistivity.

Technical composition and specific electrical resistivity of the experimental cokes: The results of the technical analysis are presented in **Table 1**.

Table 1 Technical composition of the experimental cokes after natural drying

Parameter	Unit	High-temperature coke	High-resistivity coke
Ash (Ac)		4.80	5.91
Volatiles (V)		1.24	1.97
Moisture (W)	%	3.23	1.29
Fixed carbon (C)		90.42	90.50
Total sulfur (S)		0.31	0.33

Fig. 1 shows the produced experimental coke batches: (a) high-resistivity coke and (b) high-temperature coke.



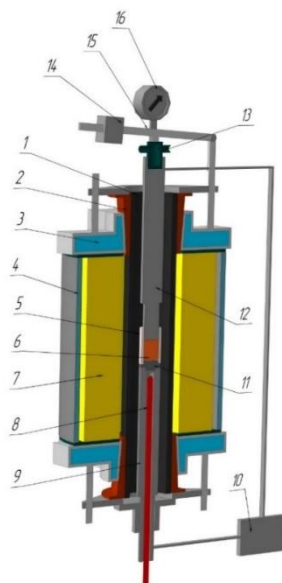
Fig. 1 The produced batches of high-resistivity coke (a) and high-temperature coke (b)

To measure and conduct a comparative analysis of the specific electrical resistivity of the obtained cokes, average samples of each material, weighing 0.5 kg, were collected. The samples were then subjected to crushing and sieving, with the 3-6 mm fraction being isolated. This procedure followed the methodology developed by Professor V.I. Zhuchkov at the Institute of Metallurgy of the Ural Branch of the Russian Academy of Sciences. This methodology enables the determination of the electrical resistivity of materials and charges at temperatures up to 1800 °C in a bulk layer. It also allows for the simultaneous fixation of the degree of softening (shrinkage).

The absolute value of specific electrical resistivity (SER) is related to the concentration of ash and volatile substances in the reducing agent. In most cases, higher concentrations of the latter correspond to larger absolute values of electrical resistivity. The literature provides numerous data on the specific electrical resistivity of various carbonaceous materials. An analysis of these data showed a significant difference in the SER values of reducing agents under normal conditions. High-temperature materials, such as the main types of coke nuts, gas cokes, and others, exhibit minimal resistivity (3-5 Ω·cm). It is observed that cokes and semicokes produced from low-metamorphosed coal grades increase the specific electrical resistivity due to their structural features.

As temperature increases, the electrical resistivity of carbonaceous materials decreases significantly. Therefore, a practical interest arises in comparing the specific electrical resistivity values of reducing agents under operational conditions, that is, at the high temperatures of the metallurgical process.

A schematic representation of the setup used to determine the electrical resistivity and thermoplastic characteristics of carbonaceous materials is provided in Fig. 2.



1 - Carbon-graphite tube; 2 - Copper compression ring; 3 - Water-cooled lid; 4 - Water-cooled housing; 5 - Alundum cup; 6 - Test material; 7 - Protective lining; 8 - Thermocouple; 9 - Lower electrode; 10 - Transformer; 11 - Graphite bottom for alundum cup; 12 - Upper electrode; 13 - Water cooling system; 14 - Weight; 15 - Lever; 16 - Dial-type internal micrometer indicator.

Fig. 2 Installation for measuring specific electrical resistivity based on the Tammann furnace (cross-section)

Two experimental coke samples were prepared for the study:

1. High-temperature coke.
2. High-resistivity coke (treated with microsilica, 15% by weight).

During the measurement of specific electrical resistivity (SER), a pressure of 0.4 kg/cm² was applied to the sample through a movable electrode. This parameter was selected to model the mechanical load on the charge column in the near-electrode zone of the ore-thermal furnace. The applied pressure allowed for the reproduction of the following: the degree of compaction of the granular layer, the area of the contact bridges, and the real contact conductivity. Thus, the chosen pressure value ensures an adequate simulation of the electrical conditions under which reducing agents operate in the furnace.

The particle size class of the materials in both cases was 3-6 mm. The temperature of the test material was continuously measured using a BP 5-20 type thermocouple. The thermocouple was placed in a hole in the cylinder through the lower electrode. The rate of temperature increase was 15-20 °C/min. This rate corresponded to the heating rate of the charge in electric furnaces.

The measurement results are presented in Table 2.

The obtained temperature dependence of the specific electrical resistivity (SER) of the experimental cokes is graphically shown in Fig. 3.

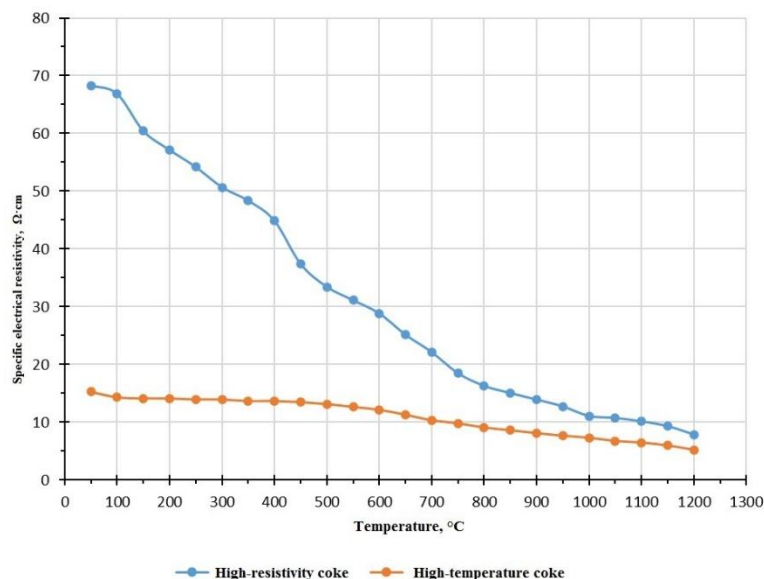


Fig. 3 Temperature dependence of the specific electrical resistivity of the experimental cokes

Table 2 Results of the measurement of specific electrical resistivity of the experimental cokes in the range of 50-1200 °C

Temperature, °C	Specific electrical resistivity, Ω·cm	
	High-temperature coke	High-resistivity coke
50	15.29	68.29
100	14.34	66.84
150	14.15	60.41
200	14.15	57.12
250	13.96	54.16
300	13.96	50.67
350	13.69	48.33
400	13.69	44.88
450	13.51	37.40
500	13.17	33.42
550	12.69	31.10
600	12.18	28.82
650	11.32	25.13
700	10.37	22.12
750	9.83	18.48
800	9.11	16.28
850	8.62	15.03
900	8.12	13.90
950	7.67	12.72
1000	7.32	11.06
1050	6.76	10.72
1100	6.48	10.13
1150	5.98	9.29
1200	5.24	7.85

As shown by the presented data, the high-resistivity coke, treated with microsilica, clearly surpasses the untreated coke in terms of specific electrical resistivity) up to a temperature of 1000°C. The high-resistivity coke was obtained by impregnating the solid carbonaceous reducing agent with an aqueous microsilica emulsion. The microsilica additive accounted for 15% by weight of the initial coal.

It is well known that, at higher temperatures, differences in the structure and mineral composition of carbonaceous materials, which affect the specific electrical resistivity, are leveled out. Consequently, electrical conductivity sharply increases. This is due to the combustion of residual aliphatic chains and the increased structural ordering in the coke.

DETERMINATION OF THE MECHANICAL PROPERTIES OF THE OBTAINED SAMPLES

The physicochemical properties of carbonaceous reducing agents are important quality characteristics for their application in ferroalloy production. The particle-size distribution and mechanical stability of the reducing agent influence the gas permeability, electrical conditions, and overall stability of furnace operation. In industrial ferroalloy furnaces, carbonaceous reducing agents with a particle size of approximately 5 (10)-25 mm are commonly used to maintain sufficient gas permeability and stable electrical and technological operating conditions.

Excessive formation of fine particles can adversely affect the gas permeability of the charge column. Although smaller particles increase the contact area between the carbonaceous reducing agent and the ore, an excessive proportion of particles smaller than 5 mm can restrict gas flow through the charge, increase dust generation, and negatively affect furnace operation. Therefore, coke mechanical

stability is an important parameter for maintaining the required particle-size distribution during handling and subsequent use in the furnace.

Carbonaceous reducing agents undergo mechanical stresses during transportation, handling, screening, charging, and movement through the furnace charge. These operations may cause particle degradation and form fine fractions. According to data reported by the Eastern Coal Chemistry Institute, the cumulative mechanical impact associated with the preparation and feeding of coke into ferroalloy furnaces can be approximated by four consecutive drops from a height of 1.8 m.

In addition to mechanical degradation during handling, carbonaceous reducing agents may undergo further fragmentation under the pressure of the charge column and during exposure to elevated temperatures inside the furnace. Consequently, evaluation of the mechanical properties of the produced coke is necessary to assess its ability to retain the required particle-size distribution under conditions representative of industrial handling and application.

The physicochemical properties of the obtained coke samples were determined using standard equipment in accordance with the relevant GOST procedures. Drop-strength and rotating-drum tests were performed to evaluate the coke's resistance to mechanical degradation and to the formation of fine fractions.

To assess the reproducibility of the experimental results, each mechanical test was performed in three independent repetitions ($n = 3$) under identical experimental conditions. For each parameter investigated, the arithmetic mean and standard deviation (SD) were calculated. The results are reported as mean $\pm$ SD. This approach was used to evaluate measurement variability and the reproducibility of the mechanical test results.

The results of the drop-strength and rotating-drum tests of the investigated carbonaceous reducing agents are summarized in **Table 3**.

Table 3 Results of the tests to determine the drop strength and the strength in the rotating drum of the investigated carbonaceous reducing agents.

Sample name	Indicators									
	Fraction yield, mm					After drop (P)				
	Initial		Before drop (without fines - 10 mm)	After drop (P)		Micum test		Irssid test		
	+ 25	- 10	+ 25	+ 25	- 10	P ₂₅	P ₁₀	i ₂₅	i ₁₀	P _{PF}
High-resistivity coke	0.7	44.4	1.3	1.0	18.8	0.8	23.0	0.4	49.9	166.7
High-temperature coke	0.3	44.9	0.5	0.7	21.1	0.4	25.3	0.3	47.4	150.0

P₂₅ - mass fraction (%) of material retained on the sieve with 25 mm openings after 100 drum revolutions;

P₁₀ - mass fraction (%) of material passing through the sieve with 10 mm openings after 100 drum revolutions;

i₂₅ - mass fraction (%) of material retained on the sieve with 25 mm openings after 500 drum revolutions;

i₁₀ - mass fraction (%) of material passing through the sieve with 10 mm openings after 500 drum revolutions;

P_{PF} - strength index characterizing the resistance of the material to fragmentation and the associated change in particle-size distribution.

The mechanical test results show differences between the two coke samples investigated. The high-temperature coke exhibited higher P₂₅ and I₂₅ values than the high-resistivity coke, indicating a slightly higher proportion of coarse particles retained after the rotating-drum tests. At the same time, the high-temperature coke showed lower P₁₀ and I₁₀ values, indicating less fine-particle formation during mechanical treatment.

The PPF values also differed between the investigated samples. Since the operational behavior of carbonaceous reducing agents in ferroalloy furnaces depends not only on their resistance to fragmentation but also on the resulting particle-size distribution, the mechanical strength indicators should be considered collectively when assessing the suitability of coke for metallurgical applications.

CONCLUSION

As a result of the conducted experimental studies, the regularities of the formation of electrical and physico-mechanical properties of high-temperature and silica-containing modified coke, obtained from the coals of JSC "Shubarkol Komir", were established.

It was established that the specific electrical resistivity of the investigated carbon materials decreased systematically with an increase in temperature across the entire studied range of 50-1200 °C. This was due to the thermoactivated growth of the electrical conductivity of the carbon matrix and the restructuring of contact bridges.

Three characteristic temperature regions of specific electrical resistivity (SER) change were identified:

I (50-400 °C) – a contact-degassing region characterized by the removal of moisture, volatile components, and the destruction of weak contact bonds.

II (400-800 °C) – a region involved intensive thermodestruction of the organic component and accompanied by the maximum rate of SER reduction.

III (above 800 °C) – a region associated with structural ordering and characterized by the growth in the degree of carbon graphitization and the convergence of the resistance values of different types of coke.

Silica surface modification significantly increased the specific electrical resistivity of coke across the entire studied temperature range.

A quantitative assessment showed the following results:

- At 50 °C, the SER increased by 4.4-4.5 times;

- In the range of 50-300 °C, it increased by 3.5-4.5 times;

- At 800 °C, it increased by 1.7-1.8 times;

- At 1200 °C, it increased by 1.4-1.5 times.

The temperature limit for the effective action of the silica modification was found to be 900-1000 °C. Above this temperature, the differences in resistivity were significantly leveled out.

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