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Thermal Engineering Calculation of Saggar for Graphite-Powder Processing with Nanoscale Coating of Carbonized Pitch as Li-Ion Battery Anode

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For the thermal treatment of powders, a special thick-walled container (a saggar) is used to ensure uniform heating of the material throughout its volume. This article presents a method of convective heating of a 1-liter laboratory reactor using flue gases from natural-gas combustion. A thermal engineering calculation and numerical modelling in COMSOL Multiphysics are conducted. The calculation involves the consideration of complex heat transfer: from the gas flow to the saggar wall, through the wall, and further into the powder layer. The problem is solved in a transient, axisymmetric formulation. Data are obtained on the temperature regimes of the material being processed and the heating rate. The simulation also enables the determination of the thermal diffusivity of the powder layer. The results are planned to be used in the design of an industrial reactor with a volume of 40 litres.

Для термічного оброблення порошків використовують спеціяльну товстостінне вмістище — саґґар, яке забезпечує рівномірне нагрівання матеріялу по всьому об'єму. У статті розглянуто спосіб конвективного нагрівання лабораторного реактора об'ємом у 1 л димовими газами від

спалювання природнього газу. Проведено теплотехнічний розрахунок і чисельне моделювання в програмному забезпеченні COMSOL Multiphysics. Розрахунок полягав у врахуванні складного теплообміну: від газового потоку до стінки саґґару та через неї, далі — у шар порошку. Завдання вирішувалося у нестаціонарній, вісесиметричній постановці. Одержано дані про температурні режими нагрівання оброблюваного матеріялу, швидкість нагрівання. Моделювання уможливило визначити температуру шару порошку. Одержані результати планується використати під час проєктування промислового реактора об'ємом у 40 л.

Key words: saggar, spheroidized graphite powder, heat transfer, thermal conductivity, thermal diffusivity, computational fluid dynamics, computer-aided design and modelling.

Ключові слова: саґґар, сфероїдизований графітовий порошок, теплопередача, теплопровідність, температуропровідність, обчислювальна гідродинаміка, комп'ютерне проєктування та моделювання.

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1. INTRODUCTION

Saggars are refractory protective containers [1] used in high-temperature thermal processing of materials to prevent contamination, oxidation, and uneven heating. Their application is especially relevant in technologies requiring high purity, stable microstructure, and reproducible properties of the final product, such as in the preparation of carbon-containing materials for the battery industry [2–5].

This study investigates the thermal treatment of high-purity graphite powder (particle size of 20 μm), modified by applying a petroleum pitch coating. The use of pitch in this case serves to create a dense carbon layer 20–40 nm thick on the surface of graphite particles, which reduces their surface porosity. For the successful application of this spheroidized-graphite powder as an anode material for lithium-ion batteries [5], it is necessary to reduce its specific surface area from 7 mI/g to 4 mI/g. This is achieved by adjusting the thickness of the deposited coating and strictly controlling the thermal regime parameters [6].

A key technological requirement is a very slow heating rate, no more than 10°C per minute, during the initial stages of pitch decomposition. This is necessary to avoid 'boiling', cracking of the coating, and the formation of surface defects. The proposed technological scheme uses heating by flue gases from natural gas combustion, while a protective atmosphere is ensured by supplying a small amount of nitrogen into the reactor. The saggar in this system en-

sures uniform temperature distribution of the processed material and protects it from contamination by combustion by-products.

The use of flue gases for heating the laboratory-industrial reactor prototype (with an internal volume of 1 litre) is justified by the intention to develop an industrial energy-efficient installation in the future (with a working saggar volume of 40 litres).

2. EXPERIMENTAL TECHNIQUE, RESULT AND DISCUSSION

The saggar is a thick-walled container (Fig. 1, *a*, *b*) equipped with a graphite tube for supplying nitrogen into a layer of spheroidized graphite powder. Figure 1, *b* shows the technical drawing of the saggar, weighing 2.2 kg, with an internal volume of 1 litre (containing 1.3 kg of powdered graphite material). The total weight of the saggar-powder system is of 3.5 kg.

The saggar (Fig. 2) is placed inside the channel of a muffle furnace with internal dimensions of 500×265×250 mm for heating with flue gases.

The drawing does not show the purged supports beneath the saggar or the lid on top, which may be integrated with the removable cover of the furnace thermal channel.

3. MODELLING AND THEORETICAL CALCULATIONS OF THE POWER REQUIRED FOR HEATING 1 KG OF GRAPHITE

This theoretical calculation serves not only for the practical selection of the burner device but also for providing initial data for computer modelling in COMSOL [7], where all further simulations were carried out.

The calculation of the thermal power of a gas burner required to heat 1 kg of graphite at a rate of 10°C/min is based on the following equation: $P = Q/t$, where Q is the amount of heat required for heating and t is the heating time.

The amount of heat Q is determined as follows: $Q = Cm\Delta T$, where C is the specific heat capacity of graphite ($C \approx 710$ J/(kg·°C)), m is the mass of graphite ($m = 1$ kg), ΔT is the temperature change.

Given the heating rate of 10°C per minute, $\Delta T = 10$ K:

1. heat required per minute: $Q = 710$ J/(kg·°C)·1 kg·10°C = 7100 J;
2. heating time: $t = 1$ min = 60 s;
3. thermal power: $P = Q/t = 7100$ J/60 s ≈ 118.33 W.

Thus, to heat 1 kg of graphite at a rate of 10°C/min, a thermal power of approximately 118.33 W is needed. For 3.5 kg of graphite, $P \approx 414$ W. To obtain 1 kWh of energy, it is necessary to burn approximately 0.1–0.12 mi of natural gas (at 100% efficiency); as-

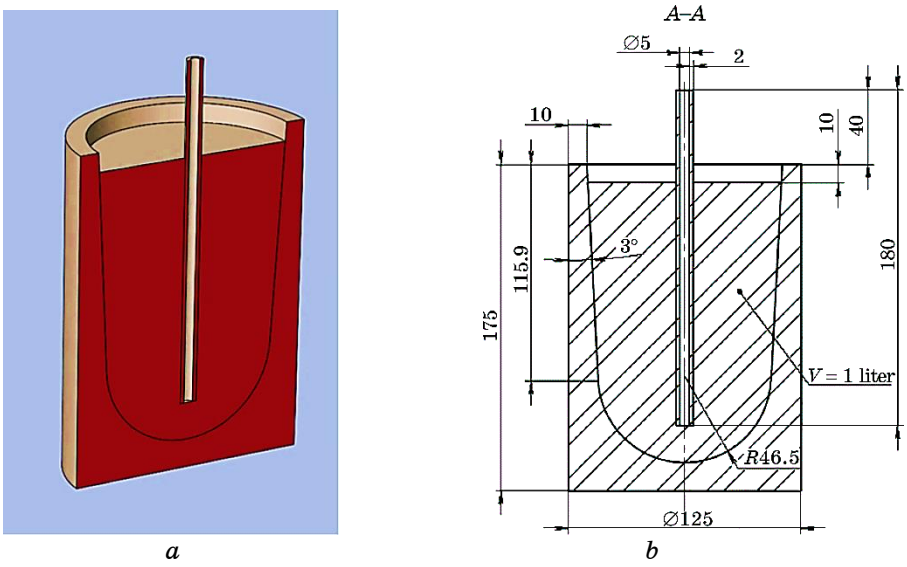


Fig. 1. Cross-section of the saggar with material: *a*—external view of the saggar; *b*—technical drawing of the saggar.

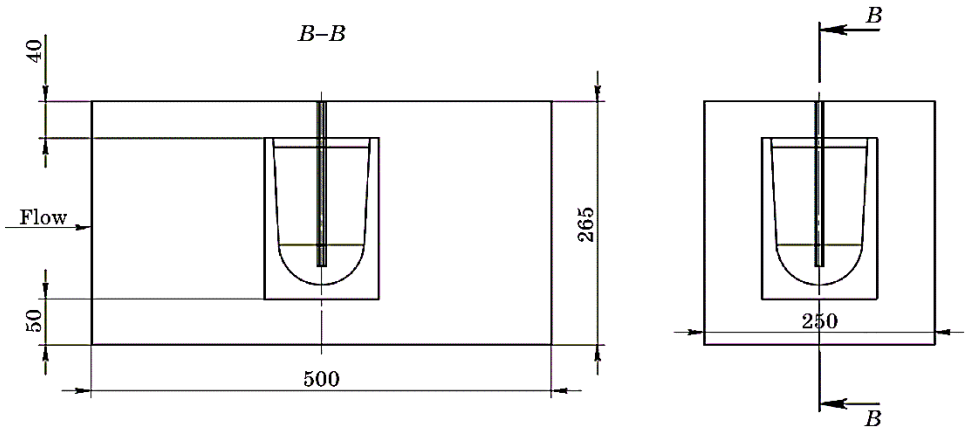


Fig. 2. Geometrical dimensions of the muffle furnace channel for heating the saggar with flue gases.

suming ≅ 90% efficiency, 0.11–0.13 mi is required.

3.1. Calculation of Flue Gas Volume from Natural Gas Combustion

Input data: volume of natural gas—0.13 m^3, flue gas temperature—900°C.

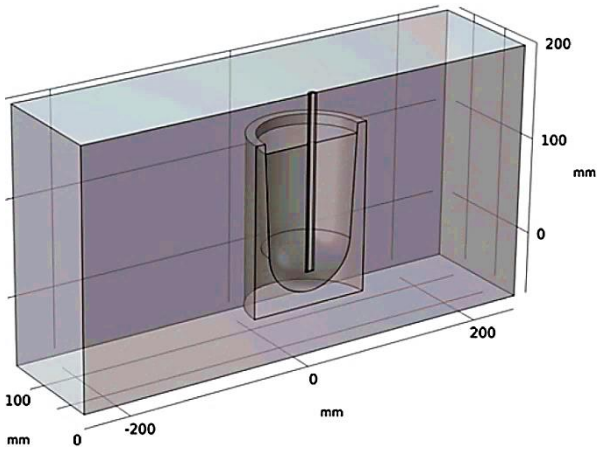


Fig. 3. Computational domain of the muffle-furnace channel for heating the saggar with flue gases.

3.1.1. Determining the Composition of Combustion Products with Assuming that Natural Gas Consists of 95% Methane (CH_4)

Combustion reaction: $CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$. Considering air (21% O_2 and 79% N_2): $CH_4 + 2(O_2 + 3.76N_2) \rightarrow CO_2 + 2H_2O + 7.52N_2$. Thus, burning 1 mi CH_4 yields: 1 mi CO_2, 2 mi H_2O (in vapour phase), 7.52 mi N_2 (from air). Total: $1 + 2 + 7.52 = 10.52$ mi of flue gases at standard conditions (0°C, 1 atm). Recalculating for 0.13 mi of natural gas: $0.13 \times 10.52 = 1.37$ mi.

Correcting for temperature of 900°C with use of ideal gas law: $V\_T = V\_0(T\_T/T\_0)$, where V_T—volume at temperature T_T, $V\_0 = 1.37$ m^3—volume at 0°C (273 K), $T\_T = 900 + 273 = 1173$ K, $T\_0 = 273$ K, $V\_1173 = 1.37 \cdot (1173/273) = 5.89$ m^3. Therefore, flue gas (f.g.) volume (for 1 kW) at 900°C is of 5.89 m^3. For power of 0.414 kW, the volume of f.g. is of 2.44 m^3. Assuming 3 kW-system, the required f.g. flow rate is of 17.67 m^3/h. The modelled axisymmetric region of the channel is shown in Fig. 3 with the following parameters: channel cross-section = 265×250 mm, area = 0.06625 m^3, gas flow speed through the section = 266.7 m/h ≈ 0.075 m/s.

3.2. Calculation of Saggar Heating by Flue Gases at 900 C

Initial data for modelling: flue gases with velocity $V = 0.075$ m/s and temperature $T_g = 900$°C are supplied from the left into the furnace channel (Fig. 4). Nitrogen is directed through a pipe to purge the powder material layer inside the saggar with velocity $V_s = 0.01$ m/s and temperature $T_0 = 20$°C; it is supplied from above through a pipe with

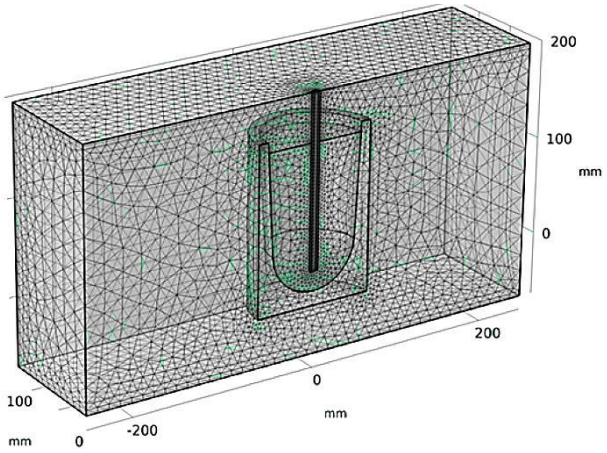
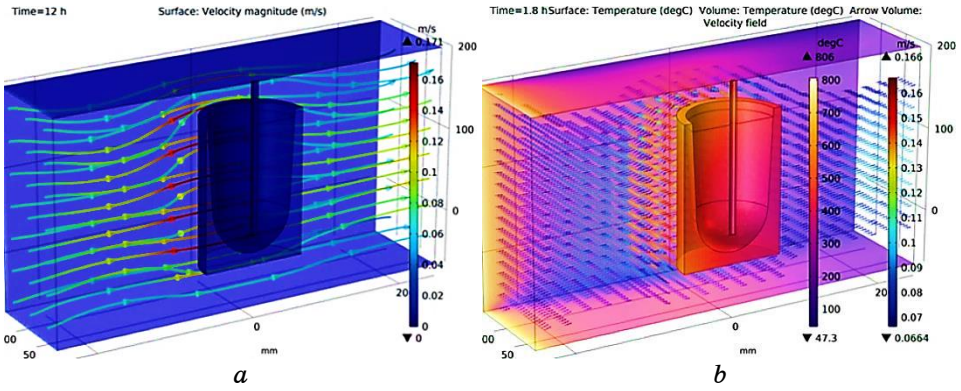


Fig. 4. Mesh of the computational domain of the muffle-furnace channel for saggar heating with flue gases, including the saggar body and the graphite powder volume.



a *b*

Fig. 5. Results of external heat-exchange simulation: *a*—velocity and flow direction; *b*—temperature distribution and flow direction.

diameter $d = 5$ mm, at a flow rate $G = 7 \cdot 10^{-4}$ mi/hour (Fig. 4).

3.3. Results and Discussions

The simulation results of the external heat exchange caused by the flow of hot flue gases are presented as streamlines of the flue gas motion (Fig. 5, *a*). The temperature distribution (isotherms) on the walls of the muffle-furnace channel and the saggar surface is shown in Fig. 5, *b*.

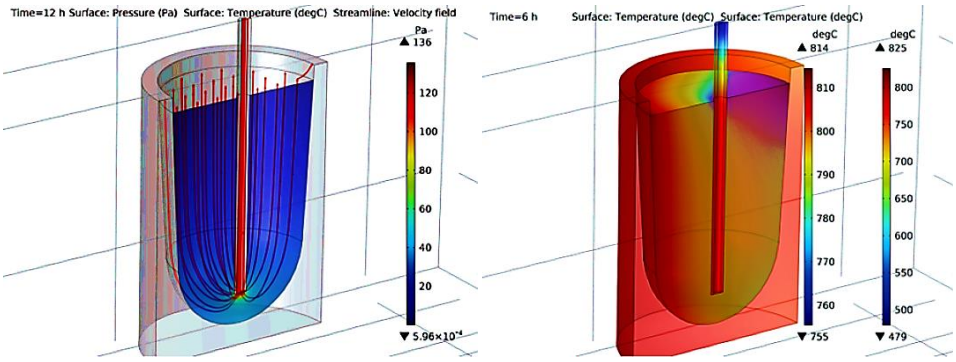
In the simulation of external heat transfer from the flue-gas flow

to the saggar surface, radiative heat exchange and convective transfer within the channel were not taken into account. However, heat losses through the external walls of the channel were considered. The physical properties of the powdered graphite layer are presented in Table 1.

The permeability of the graphite powder layer was calculated using the Ergun equation: $k = \varepsilon^2 d_p^2 (1-\varepsilon)^{-2} / 150$. At a porosity of 40%, the permeability (according to Ergun) is approximately of $k \approx 4.74 \cdot 10^{-13}$ m^3. The nitrogen flow through the porous material layer (Fig. 6) was simulated using the specialized Brinkman module.

TABLE 1. Physical properties of the powdered graphite layer.

Particle diameter, μm	Particle shape	Density, g/cmi	Porosity, %	Bulk density, g/cmi	Thermal conductivity, W/(m·K)	Specific heat capacity, J/(kg·K)	Layer permeability, mI
20	Spherical	2.2	40–50	1.1–1.3	80–150 (true), 1–10 (powder)	700–900	$k \approx 4.74 \cdot 10^{-13}$



a *b*

Fig. 6. Results of modelling the internal space of the saggar: *a*—nitrogen pressure generated by the layer of porous graphite material; *b*—temperature distribution in the saggar and graphite powder.

For modelling the external and internal heat exchange through the porous nitrogen body (Fig. 6), a Multiphysics simulation module was used, combining the Brinkman and Heat Transfer modules for porous media with external flow. The coupled solution of the corresponding differential equations allowed taking into account the mutual influence of hydrodynamic and thermal flows passing through different physical materials and media. The time-dependent dynamics of the average and minimum temperatures of the porous graphite material are presented in the graph (Fig. 7).

The dependences indicate that the main heating of the powdered material occurs within 4–6 hours of processing. Afterwards, the

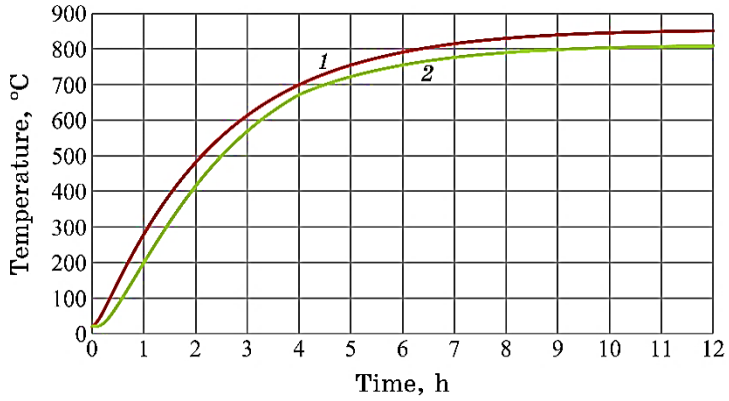


Fig. 7. Temperature of the porous graphite material inside the saggar. *1*—Temperature_Ave (degC); *2*—Temperature_Min (degC).

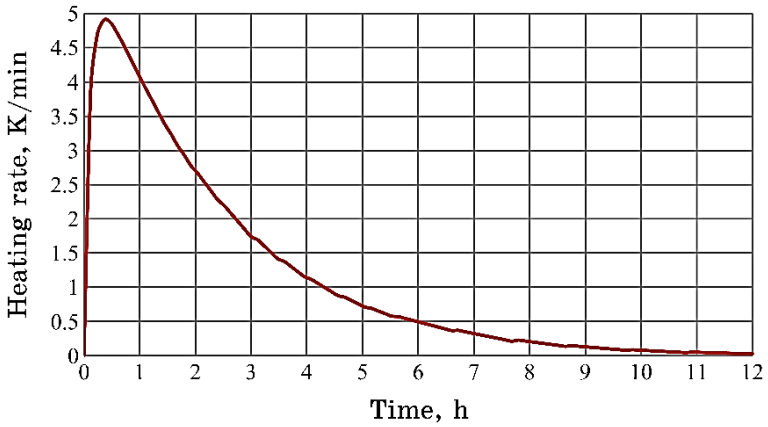


Fig. 8. Average heating rate of the porous material for various heat carrier temperatures.

material's temperature exponentially approaches the temperature of the heat carrier. It can be approximately considered that the temperature change ceases after 12 hours. It is shown that the heating time of the processed material does not significantly depend on the heat carrier temperature under the given conditions.

The average heating rate (Fig. 8) under the given conditions reliably does not exceed the established technological limit of 10 degrees per minute.

4. THERMAL EFFECTS UPON THE INTERACTION OF PULSED RADIATION WITH THE MATERIAL

The thermal diffusivity of powdered graphite coated with a layer of carbonized petroleum pitch was determined using a pulsed thermal diffusivity measurement method. This method is non-destructive and contactless. The developed technique requires special sample preparation—pressing the powder into a pellet. The pressing force is minimal to ensure that the sample is not destroyed and, at the same time, does not significantly differ from the structure of the powder processed in the saggar.

The front surface of the sample is irradiated with a short light pulse (Fig. 9, *a*) in the visible range (pulse duration of 100 ms), generating a primary radiation flux.

The absorbed portion of the energy is converted into heat, causing local heating that gradually spreads throughout the volume of the material. Part of the heat flux reaches the backside of the sam-

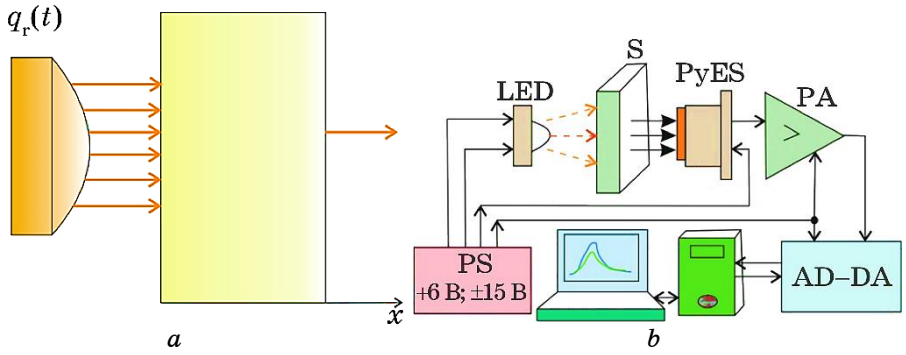


Fig. 9. Scheme and working principle of the setup for measuring thermal diffusivity: *a*—model of the heat flux redistribution process through the sample; *b*—block diagram of the pulsed thermal diffusivity measurement method: LED diode (GREE-XML); S—graphite material sample; PyEs—pyroelectric sensor (IRA-E700ST0); PA—amplifier (Gain = 10.000); AD-DA—ADC-DAC unit (±10 V, 12-bit resolution, 2 channels); SPS—computer.

ple, where a secondary infrared radiation flux is generated. This secondary flux, delayed in time relative to the primary one due to the finite speed of thermal diffusion, is detected by an IR sensor. When using a pyroelectric sensor as the IR detector, it is not necessary to know precisely the energy of the incident and absorbed radiation, the absorption coefficient of the front surface, the emissivity of the back surface, as well as the temperature curve of heating and the sensitivity parameters of the sensor [8, 9].

The block diagram of the measurement system is shown in Fig. 9, *b*. In this setup, a light-emitting diode (LED) was used as the heat radiation source, and a pyroelectric sensor (PyES) was employed to register the thermal response. This method allows effective and reliable determination of the thermal diffusivity of graphite materials under pulsed heating conditions.

Thermal diffusivity is critically important in modelling tasks, as it directly affects the accuracy of numerical calculations of temperature fields. Moreover, during scale-up from laboratory to industrial volume, thermal diffusivity acts as one of the key variables enabling the transfer of thermal regime parameters without loss of heat treatment quality. Therefore, accurate evaluation of the thermal diffusivity of the studied graphite powder is an essential step in developing and scaling an effective technology for producing anodic material of the required quality.

The dynamics of thermal diffusivity changes in the porous graphite material are presented in the graphs (Fig. 10, *a*, *b*).

The thermal diffusivity of graphite powder depends on several factors, including: particle size and shape, packing density, presence of impurities in the powder, temperature, anisotropy of graphite (layered structure), as shown in Table 2.

Thus, the thermal diffusivity of the graphite powder is approximately as follows: $a \approx 0.001–0.02$ m^2/s. For the studied graphite powder, the dependence of thermal diffusivity (Fig. 10, *b*) on the temperature of the porous graphite material appears almost linear, ranging from $0.2e^{-4}$ m^2/s at the initial 20°C to $2e^{-4}$ m^2/s at 900°C.

5. CONCLUSIONS

A model of thermal interaction in the system ‘flue gases–saggar–graphite powder’ was developed and implemented, taking into account the porous structure features of the spheroidized powdered graphite material under protective atmosphere conditions. Numerical modelling allowed obtaining data on the temperature characteristics of the spheroidized powdered graphite material over time, including average and maximum temperatures, as well as heating rates that are critically important for preserving the structure of

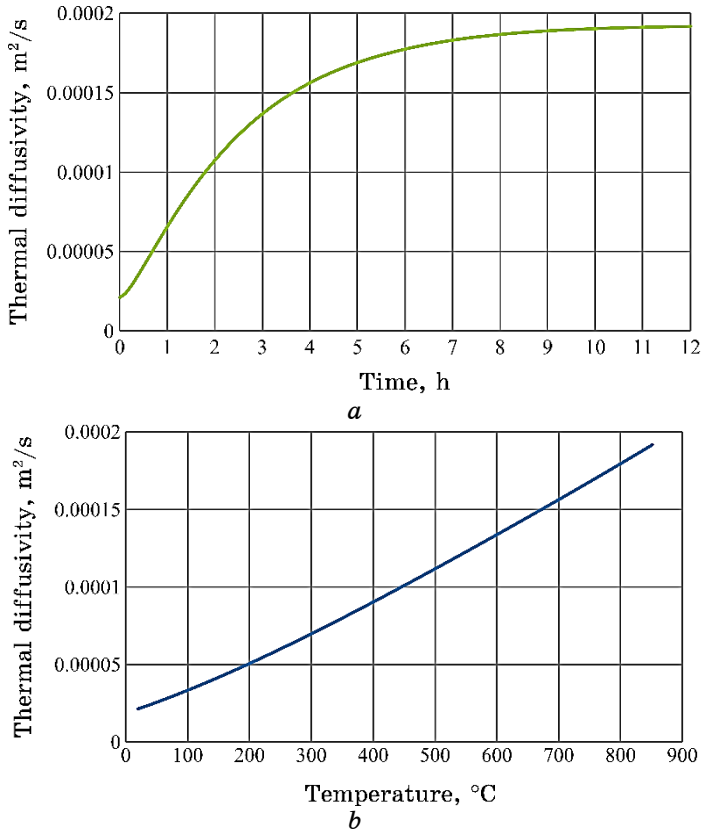


Fig. 10. Changes in the thermal diffusivity of porous graphite material: *a*—depending on time; *b*—depending on temperature.

TABLE 2. Thermophysical properties of graphite powder.

Thermal conductivity, W/(m·K)	Density (compacted), kg/mi	Specific heat capacity, J/(kg·K)	Thermal diffusivity, m^3/s
1–10	500–1200	700–900	0.001–0.02

the pitch coating.

A device for the pulse method of measuring the thermal diffusivity of materials of different nature with data recording on a PC was developed. Measurements of thermal diffusivity were conducted on samples of spheroidized powdered graphite material. The calculated values of thermal diffusivity of the graphite powder correlate well with known literature [10] and experimental data, confirming the correctness of the applied model. It was established that, at the given heat carrier temperature and the corresponding flue gas flow

rate, the heating rate of the graphite powder does not exceed the established technological limit of 10°C per minute. The use of a compact laboratory-industrial installation with a volume of 1 litre is justified as a stage of preliminary validation of thermal calculations, the results of which can be scaled for the design of an industrial saggar with a volume of 40 litres. The developed methodology can be applied for calculating and optimizing thermal regimes in similar heat treatment processes of porous materials with higher requirements for uniformity and property control.

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REFERENCES

1. J. Zhang, Z. Wang, and M. Li, *Journal of Materials Processing Technology*, **288**: 116856 (2021); https://doi.org/10.1016/j.est.2022.104408
2. E. Strativnov, A. Khovavko, and G. Nie, *Appl. Nanosci.*, **13**: 7531 (2023); https://doi.org/10.1007/s13204-023-02904-4
3. E. Strativnov, A. Khovavko, and N. Guachao, *Appl. Nanosci.*, **12**: 2791 (2022); https://doi.org/10.1007/s13204-022-02589-1
4. J. Waetzig, T. Hutzler, and E. Zschippang, *International Journal of Applied Ceramic Technology*, **22**, Iss. 1: e14897 (2025); https://doi.org/10.1111/ijac.14897
5. Xu PengTao, Yang JiXiang, Wang KeSai, Zhou Zhen, and Shen PanWen, *Chines Science Bulletin*, **57**, No. 23: 2948 (2012); https://doi.org/10.1007/s11434-012-5121-3
6. *COMSOL Multiphysics*® (2018). *Heat Transfer Module User's Guide. COMSOL 5.4*; https://doc.comsol.com/5.4/doc/com.comsol.help.heat/HeatTransferModuleUsersGuide.pdf
7. B.-R. Kim, Ji-Ra Kim, and Ji-Sun Im, *Materials*, **15**, No. 13: 4713 (2022); https://doi.org/10.3390/ma15134713
8. Iryna Trosnikova, Anatolii Minitskyi, Petro Loboda, and Maksym Barabash, *Results in Materials*, **23**: 100620 (2024); https://doi.org/10.1016/j.rinma.2024.100620
9. Alexander Khovavko, Eugene Strativnov, Denis Filonenko, Andrii Nebesnyi, Olexiy Sviatenko, Angela Piatova, and Maksym Barabash, *Carbon Nanosructured Materials: Synthesis and Application* (Springer: 2024); https://doi.org/10.1007/978-3-031-64121-3
10. Wenchan Zhang, Hua Dong, Yongchun Wang, and Jingkui Zhang, *International Journal of Thermophysics*, **38**: Article number 120 (2017); https://doi.org/10.1007/s10765-017-2261-3