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Determination and comparison of hydrogen content in industrial graphite materials

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The study of hydrogen content in graphite materials is relevant due to its potential impact on the physical and mechanical properties of these structural materials operating at extremely high temperatures. The objective of this study was to directly measure hydrogen content in carbon materials (samples up to 1.0 g) and develop a simple method for its determination in industrial graphite materials. The hydrogen content in graphite, pyrolytic graphite, and carbon-carbon composite material (CCCM) was determined using a setup including a vacuum chamber, laser, and gas dehydrator (silica gel). It was found that the materials contain 3–5 at.% (0.3–0.45 wt.%) hydrogen.

Keywords: hydrogen, graphite, laser, silica gel.

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Introduction

Graphite and carbon-carbon composite materials are widely used as part of refractory structures [1, 2], structures and parts resistant to the action of high-temperature gas [3, 4] and plasma flows [5, 6, 7].

The problem of studying the hydrogen content in graphite materials is relevant due to the possible influence of hydrogen on the physicomaterial properties of such multifunctional structural materials, which generally operate under conditions of extremely high temperatures.

In recent years, the greatest number of works devoted to the study of the influence of hydrogen on the properties of graphite have had a purely practical orientation associated with the use of graphite for protecting the first wall of thermonuclear reactors [5–8]. In most studies, it is usually assumed that the process of interaction of hydrogen atoms with graphite is purely surface-related and limited to the temperature region below 1500 K, while the possible

hydrogen content in the bulk of graphite materials is most often neglected [5].

The objective of this work is to develop a simple methodology for determining the hydrogen content in various industrial types of graphite materials, averaged over the sample volume and to compare the obtained results.

At the initial stage of the work, an elemental composition analyzer CHNS "Eurovector" EA-3000 was used to determine the hydrogen content, making it possible to determine the content of nitrogen, carbon, hydrogen, nitrogen, and sulfur contents in materials.

The principle of operation of the analyzer is based on the combustion of a sub-milligram or few-milligram sample of the analyzed substance in pure oxygen.

Three series of measurements were performed. Their results are presented in Table 1. The probable error of the results is about 0.1–0.2% according to the data of the developer of the elemental composition analyzer CHNS "Eurovector" EA-3000.

Table 1

Hydrogen content (wt.%) in samples of different graphite materials according to three series of measurements

Material	1st series		2nd series		3rd series		Average
Graphite MPG-7	0.51	0.58	0.39	0.32	0.39	0.33	0.42
Pyrolytic graphite	0.37	0.41	0.41	0.35	0.36	0.45	0.39
UUKM	0.46	0.36	0.57	0.51	0.42	0.45	0.46

As can be seen, the measurements confirmed the presence of hydrogen in graphite materials and gave its average content of about 0.42 wt.% with a scatter of values of about 0.1%. At the same time, no significant change in the hydrogen content in different materials was revealed. The disadvantage of the performed measurements is the small mass of the studied materials (several mg), which under conditions of their significant spatial non-uniformity (especially in UUKM), can cause an increased scatter of results.

The obtained data are qualitatively consistent with the results of analogous measurements performed at NIIgrafit [9], where for a material of the UUKM type after impregnation and carbonization a value of 1.1 wt.% was obtained, and after high-temperature treatment (HTT) at 2500 K – 0.3 wt.%.

The aim of the present work is the direct measurement of the hydrogen content in carbon materials with sample masses up to 1.0 gram.

Methodology of conducting the experiments

The determination of the hydrogen content in graphite materials consists in burning the sample in an oxygen atmosphere with the subsequent determination of the amount of water vapor in the combustion products.

Fig. 1 presents a diagram of the experimental setup for measuring the hydrogen content in carbon materials.

To absorb water vapor (to dry the gas mixture formed after combustion of the sample in oxygen), silica gel according to GOST 3956-76 in the form of granules with a diameter of 2–4 mm, previously subjected to heat treatment at a temperature of 150–200 °C, was used. Silica gel is capable of drying various gases (air, oxygen, carbon dioxide, nitrogen, hydrogen, argon, etc.) at atmospheric pressure. It was preliminarily

verified that 1 g of silica gel is capable of absorbing up to 50–60 mg of vapor to saturation, that the time of its saturation in open air with a humidity of 40% is 4–6 hours, and also that all the additional water specially introduced into the working chamber (5–15 mg) upon reproduction of the working cycle gave a corresponding increase in the mass of the silica gel.

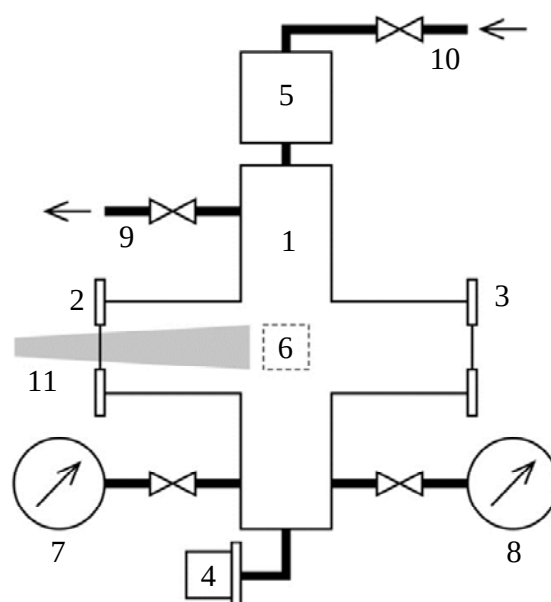


Fig. 1. Experimental setup diagram: 1 – vacuum chamber; 2, 3 – optical windows; 4 – tube with a flange for silica gel; 5 – tube with a flange for a weighed portion of water; 6 – sample; 7 – valve and manometer; 8 – valve and vacuum gauge; 9 – valve for connection to the pump; 10 – valve for oxygen supply; 11 – laser radiation

The sequence of conducting the experimental studies was as follows.

1. A sample of graphite material with dimensions of 40×6×2 mm and a weight from 0.3 to 1.0 g was installed in the vacuum chamber.

2. The stainless steel vacuum chamber with a volume of about 250 cm³, after evacuation, was filled with oxygen to a pressure of 0.3–0.7 MPa.

3. Silica gel with a mass of 1–2 g in the form of granules was placed in a bag made of metal mesh, which was located in a tube (with a

diameter of 30 mm and a length of about 50 mm) docked to the vacuum chamber.

4. The process of burning the graphite material was ensured by the action of laser radiation with a power of 400 W for 10 seconds.

5. After combustion of part of the sample (until the oxygen was burned out), the chamber was dried for the required time (usually 12–48 hours), after which the pressure in the chamber was reduced to atmospheric. For each pressure value, the required drying time must be determined experimentally by ensuring complete absorption of the additionally introduced known amount of water.

6. The chamber is opened, and the bag with silica gel is quickly removed.

7. The silica gel is weighed every minute for 15–30 minutes in order to determine as accurately as possible the initial mass of the silica gel at the moment of its removal by extrapolating the obtained growing dependence (due to absorption of vapor from the air) of mass on the

time spent in air to the time of removal of the bag.

8. The remainder of the partially burned sample of material is removed from the chamber and weighed.

As direct control measurements have shown, a correction must be introduced into the obtained results for the absorption of moisture from the air by the silica gel during the time between its first weighing and installation in the sealed volume of the chamber, and then between removal from the sealed volume and weighing. This time is about one minute, during which the silica gel gains about 1 mg of moisture from the ambient air of the room (with a relative humidity of about 40%).

Characteristic quantities: the burned mass of carbon is 0.35–0.45 g, and the increase in the mass of silica gel is 8–13 mg.

The general view of the setup for measuring hydrogen in graphite materials is presented in Fig. 2.



a)



b)



c)

Fig. 2. Setup for measuring hydrogen in graphite materials: a) – initial state; b) – moment of laser ignition; c) – combustion of the sample

Discussion of the results

The obtained results are presented in Table 2.

It can be seen that the hydrogen content in different samples obtained by cutting from different places of one block of material is substantially different.

Table 2

Results of measurements of hydrogen content in wt. %

Material	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Mean
MPG-7	0.32	0.36	0.26	0.31	–	0.31 ± 0.03
UUKM	0.36	0.45	0.41	0.38	0.38	0.4 ± 0.03

These results are consistent with those obtained earlier and for the first time indicate a possibly higher hydrogen content in UUKM, which undergoes multiple impregnation with pitch with subsequent carbonizing firings and, what is especially important, multiple high-temperature graphitizing firings, which apparently contribute not to the removal of hydrogen from the material, but, on the contrary, to its saturation with it.

The mass-spectrometric studies of the composition of graphite materials performed by the authors of [10], in our opinion, also indicate a significant (about 0.5 wt.%) hydrogen content in graphite and its presence in vapor phase in a bound state (as part of hydrocarbons).

It is significant that not once was a hydrogen content greater than 0.51 wt.% recorded.

Conclusion

Thus, according to the data of the work [9], the measurements on the "Eurovector" EC-3000, mass spectroscopy data [10], and the results obtained in the present work, industrial graphite materials always contain about 3–5 at % (0.3–0.45 wt.%) hydrogen. This is obviously due to the technology of their production (either upon saturation with pitch, or in a hydrogen atmosphere during gas-phase deposition).

The mastering of operational monitoring of the hydrogen content in industrial graphite materials opens up the possibility of a comprehensive study of the influence of this element on the physicomachanical characteristics of materials, as well as on their behavior at high temperatures.

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