RESEARCH INTO RECYCLING OF NICKEL-COBALT-CONTAINING METALLURGICAL WASTES BY THE ECOLOGICALLY-SAFE TECHNIQUE OF HYDROGEN REDUCTION

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Дослідженно кінетику водневого відновлення окалини нікель-кобальтвмісного прецизійного сплаву. Цільовий продукт металізації після відновлення при 1273 К мав губчасту мікроструктуру, переважно складався з твердого розчину атомів Ni та Co в $\gamma-Fe$. Також виявлено залишок Fe_3O_4 та FeO. Дослідно-промислові випробування підтвердили ефективність використання нової легуючої добавки з паралельною утилізацією техногенних відходів

Ключові слова: окалина, прецизійний сплав, водневе відновлення, фазовий аналіз, мікроструктура, ресурсозбереження, легування

Исследована кинетика водородного восстановления окалины никель-кобальтсодержащего прецизионного сплава. Целевой продукт металлизации после восстановления при 1273 К имел губчатую микроструктуру, преимущественно состоял из твердого раствора атомов Ni и Co в $\gamma-Fe$. Также выявлен остаток Fe_3O_4 и FeO. Опытно-промышленные испытания подтвердили эффективность использования новой легирующей добавки с параллельной утилизацией техногенных отходов

Ключевые слова: окалина, прецизионный сплав, водородное восстановление, фазовый анализ, микроструктура, ресурсосбережение, легирование

1. Introduction

Recent years have seen increased demand for steel, alloyed with rare and refractory elements, in particular, Ni and Co. At the same time, prices for the respective alloying materials in the global market demonstrate a trend to grow [1].

One of the alternative sources for obtaining the Ni and Co alloying materials is the recycling and bringing the al-

loyed technogenic waste back to production. These include the scale of nickel-cobalt containing precision alloys. The volume of its formation is relatively high and accounts for 3–4 % by weight of smelting; when producing small-section billets – 9–12 % by weight. The specific feature of this type of waste is a high alloying degree. This necessitates taking into consideration the complex nature of physical-chemical interaction between elements when designing technological conditions for recycling.

Processing techniques may include the utilization of solid-phase reduction with carbon or hydrogen, as well as reduction melting in the system of rare-phase reactions. The application of solid-phase reduction is safer ecologically since it requires lower temperatures and energy consumption. In other words, it ensures a relatively smaller emission of gaseous products of reactions into the environment.

Therefore, there is a relevant task of resource and energy saving with reduced losses of Ni and Co when recycling and using the scale of precision alloys in steelmaking industry. A strategic direction for solving this problem is to develop an understanding of the reduction mechanism of oxide nickel-cobalt containing raw materials. Along with this, there is a significant task to improve ecological safety of the environment in industrial regions. Achieving positive results in addressing this challenge is possible by replacing the resource- and energy-intensive smelting technologies of refractory alloying materials with modern methods of solid-phase metallization in powder metallurgy.

2. Literature review and problem statement

In powder metallurgy, one can highlight carbon-thermal and hydrogen technique for the reduction of oxide technogenic raw materials from the existing list of solid-phase reduction. Carbon-thermal is simpler technologically and more attractive economically. The authors of paper [2] conducted a thermodynamic modeling of chemical and phase transformations in the system Fe_2O_3–NiO–CoO–C at temperatures of 573–1773 K. It was determined that Fe from the oxide mostly passes into α–Fe. The degree of transition of Fe increases from 28.9 % at 1173 K to 99.05 % at 1773 K. The degree of transition of Ni and Co during reduction from the oxides to metallic phases approaches 100 % in a temperature range of 573–1273 K. Further temperature increase to 1773 K leads to a noticeable increase in the losses of Ni and Co through sublimation into the gas phase and to a decrease in the degree of extraction of these elements. The reduction products, however, may contain residual carbon associated with the oxycarbide and carbide compounds [3]. The formation of Fe carbides is confirmed [4] in parallel with the reduction in the process of carbon-thermal treatment of oxide raw materials. Research into thermodynamic regularities of carbon-thermal reduction in the system Co–O–C [5] indicates that an increase in temperature results in primary transition of higher oxides into lower oxides. Article [5] also argues about the probability of reaction of carbide formation with the reduction to free Co. In turn, nickel-cobalt containing precision alloys such as 29NK [6, 7], possess strict restrictions for carbon. Given this, the use of carbon-thermal treatment for the reduction and recycling of scale of precision alloys may cause technological complications, while being impossible at all in some cases. It is more rational in this case to choose the hydrogen reduction.

Paper [8] explored thermodynamic patterns of reduction in the system Ni–Co–O–H in a temperature range of 300–2000 K. The authors determined a thermodynamic probability of reducing the Ni and Co oxides that can be present as components of the scale of precision alloys. In this case, NiO has a higher susceptibility to the hydrogen reduction than CoO.

Study into NiO reduction at temperatures of 543–1593 K was carried out by authors of article [9]. It was determined that at 543–773 K, reaction products were a mixture of Ni and NiO. At 1173–1593 K, the rate of reactions significantly increased resulting in full reduction to Ni. The structure is sintered, with fine pores. Taking the above into consideration, it is more expedient to choose a different interval of relatively high temperatures for the experiments.

Authors of paper [10] investigated hydrogen reduction of iron oxides, which can be components of the scale of precision alloys. It was established that the full reduction of relatively small samples of Fe_2O_3 to Fe at heating rate of 500 °C/h proceeds until reaching a temperature of 420 °C. Increasing the density of the material of samples leads to a significant decrease in the rate of reduction – heat treatment temperatures reached values above 900 °C. It testifies to the essential importance of gas exchange between reaction products and the inhibition of the latter with an increase of the density of samples that should be considered when reducing the scale of precision alloys.

Solid-phase reduction of scale with hydrogen with obtaining iron powder is examined in article [11]. The base of the scale consisted of Fe_2O_3 and FeO. Full reduction was achieved after treatment at 1,000 °C. The temperature of reduction at 1,100 °C led to intensive sintering of particles. In other words, similar patterns can manifest themselves during reduction of the oxide iron-containing part of the scale of precision alloys. This should be taken into consideration when conducting experiments.

An analysis of the scientific literature that we conducted reveals the availability of significant results in the studies into processes of hydrogen reduction of Fe, Ni and Co using separately taken oxides as an example [8–10]. There are also achievements related to the non-alloyed iron-containing oxide technogenic waste [11]. However, the mechanism of the course of the processes during hydrogen reduction of complex-alloyed oxide technogenic raw materials, such as the scale of nickel cobalt-containing precision alloys, was not studied sufficiently enough. Parallel participation in the process of oxide compounds of Ni, Co and Fe, as well as other impurities in the composition of highly-alloyed scale can significantly affect the course of reduction. This may also affect the microstructure and phase composition of the end products of metallization. Research in this direction can reduce the loss of Co and Ni through the sublimation of oxide compounds during reduction of technogenic raw materials and reuse of metallized alloying additive. Based on the above mentioned, it is a relevant task to comprehensively study the kinetics of hydrogen reduction of the scale of a nickel-cobalt containing precision alloy, as well as the phase composition and microstructure of metallization products.

3. The aim and objectives of the study

The aim of present work was to study physical-chemical patterns during recycling of the scale of nickel cobalt-con-

taining precision alloys using the method of hydrogen reduction. This is necessary in order to identify parameters that reduce the loss of Co and Ni during processing of oxide technogenic waste and when using the obtained metallized alloying additives in steelmaking industry.

To achieve the set aim, the following tasks had to be solved:

– to explore kinetic patterns of hydrogen reduction of the scale of a nickel-cobalt containing precision alloy at different temperatures;

– to examine phase composition and microstructure of the metallized scale of a nickel-cobalt containing precision alloy with varying degrees of reduction, in order to determine the mechanism of transformations course during heat treatment.

4. Materials and methods to study the kinetics of reduction and the products of metallization of the scale of a nickel-cobalt containing precision alloy

4. 1. The examined materials and equipment used in the experiment

The starting raw material is the precision alloy's scale of the following chemical composition, % by weight: C – 0.023; Si – 0.230; Mn – 0.305; S – 0.015; P – 0.015; Cr – 0.076; Ni – 21.990; Co – 13.390; Cu – 0.150; Al – 0.150; Ti – 0.076; O – 24.400; Fe – the remainder.

The study into kinetics of reduction was performed at the universal thermogravimetric installation of the flow-through type (Fig. 1).
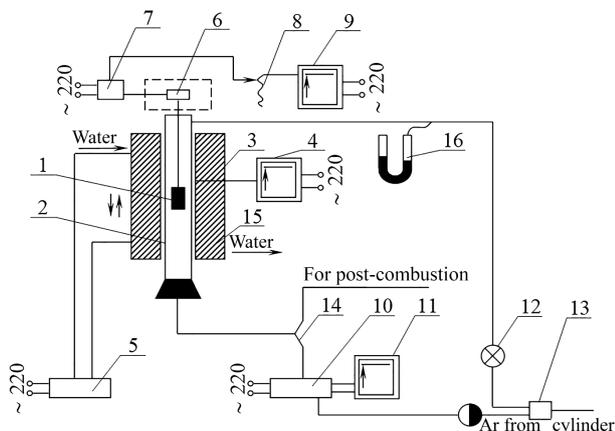

Fig. 1. Schematic of the thermogravimetric installation:
1 — sample; 2 —reaction tube; 3 — thermocouple;
4 — potentiometer KSP2-003; 5 — furnace control unit;
6 — mechanotron 6MH8B; 7 — electronic unit;
8 — regulatory supports MKL-49; 9 — potentiometer KSP-4;
10 — chromatograph LHM-80; 11 — potentiometer KSP-4;
12 — regulating valves; 13 — gas reducer; 14 — tee;
15 — resistance furnace; 16 — air pressure gauge

We used drained H_2 as a reducer, which was obtained from the hydrogen generator "Hydrogen-1" (Ukraine) and fed to the reaction zones through regulating valves (Fig. 1). Protective gas for blowing the system is Ar, in line with GOST 10157-79.

X-ray phase analysis of the samples was carried out using the diffractometer "DRON-6" (Russia).

Images of the microstructure of samples were acquired using the raster electron microscope "REM-106" (Ukraine).

4. 2. Procedure for conducting experiments and determining the indicators of sample properties

The temperature interval of kinetic studies is 673–1573 K; period is 180 minutes; hydrogen consumption is 9 l/h; linear gas flow rate is 8×10^{-3} m/s. During experiments, we continuously measured changes in the mass of the samples. To check for the absence of sublimation, we calculated the mass of oxygen in the scale, which reacted with hydrogen, and compared with the change in the mass of the sample over the duration of experiment.

The samples were exposed, for performing a phase analysis and conducting the study into microstructure, to isothermal treatment at a temperature of 1273 K in the hydrogen atmosphere. The experiments were stopped at timed intervals to obtain a product with varying degrees of reduction.

The phase composition of the examined samples was determined employing a method of X-ray structural analysis using the monochromatic radiation of Co $K_α$ ($λ$=0.178897 Å) with a Fe filter. The measurements were performed at a voltage on the tube of U=30 kV and anode current I=10 mA. Composition of the phases was determined applying the software package PDWin 2.0 (Russia).

Study of the samples' microstructure was carried out at accelerating voltage 20 kV and current of the electron probe 52–96 µA. A working distance to the examined surface was 10.5–10.7 mm.

5. Results of research into hydrogen reduction of the scale of a nickel-cobalt containing precision alloy

Treatment at a temperature of 673 K yielded the degree of reduction of 12 % (Fig. 2, Curve 1). A further increase in the temperature of thermal treatment to indicators 723 K, 773 K, and 823 K was characterized by an increase in the degree of reduction of the resulting product to 16 %, 23 %, and 38 %, respectively (Fig. 2, Curves 2–4).

Treatment at 1273 K provided a faster reduction rate. At min. 100, the degree of reduction was 94 % (Fig. 2, Curve 5). The process subsequently proceeded in the diffusion region with a clearly pronounced decrease in the rate of reduction. Finally, we achieved the degree of reduction of 99 %.

At 1423 K, 1473 K, 1523 K, 1573 (Fig. 2, Curves 6–9), the initial period of oxygen removal proceeded at a relatively high speed. The process subsequently proceeded with much lower speed compared to the results obtained in the previous case. Over 180 minutes of thermal treatment, the degree of reduction was 80 %, 72 %, 66 %, and 61 %, respectively.

The mass of removed oxygen in all experiments was practically equal to a decrease in the mass of the sample during experiment, which testifies to the absence of sublimation of alloying elements under conditions of conducting the experiments.

The scale is represented by Fe_3O_4, Fe_2O_3, FeO, probably with the atoms of Ni and Co dissolved there (Fig. 3). At 12 % of reduction we observed the emergence of a FeNi phase and the disappearance of a Fe_2O_3 manifestation. At the degree of reduction in an interval from 23 % to 92 %, the diffractograms showed a phase of γ–Fe.
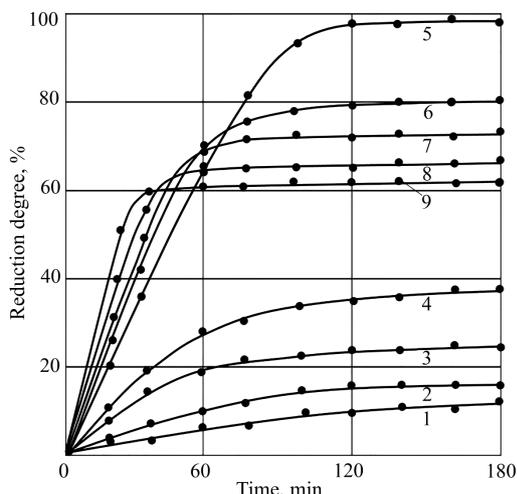
Fig. 2. Kinetics of hydrogen reduction of the scale of a nickel-cobalt containing precision alloy at different temperatures: 1 — 673 K; 2 — 723 K; 3 — 773 K; 4 — 823 K; 5 — 1223 K; 6 — 1423 K; 7 — 1473 K; 8 — 1523 K; 9 — 1573 K
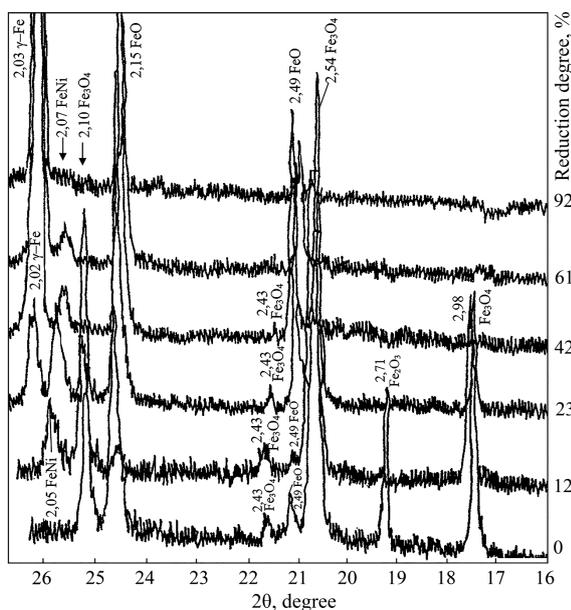

Fig. 3. Sections of diffractograms of samples of the scale of a precision nickel-cobalt containing alloy with varying degrees of reduction. Figures next to the diffraction maxima correspond to parameters of the crystal lattice
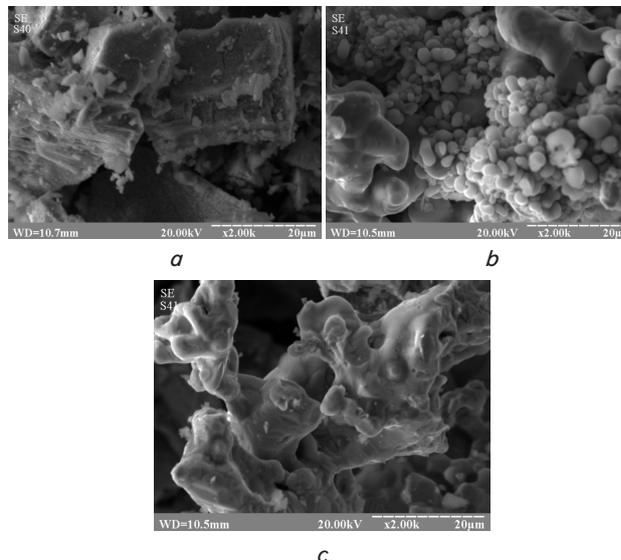

Fig. 4. Images of the microstructure of metallized scale of a nickel-cobalt containing precision alloy after hydrogen reduction at different temperatures, with magnification ×2000: a — 673 K, b — 1273 K, c — 1573 K

6. Discussion of results of research into hydrogen reduction of the scale of a nickel-cobalt containing precision alloy

The kinetic research conducted (Fig. 2) indicates that treatment at 673–823 K does not provide high enough degree of reduction. Of practical interest is the heat treatment at 1273 K with achieving a degree of reduction of 99 %. This agrees well with the results of papers [10, 11] whose authors under similar conditions attained the most effective indicators of reduction. Increasing the temperature of treatment from 1423 K to 1573 K is accompanied by a decrease in the degree of reduction of samples from 80 % to 61 %, respectively. This may be due to the formation of surface "block-layers" of the sintered particles, clearly confirmed by microscopic studies (Fig. 4). The result of this is the complicated gas exchange that shifts the reduction process from the kinetic to the diffusion region. It becomes apparent that a further rise in temperature plays a negative role for achieving improved rate and degree of reduction. Similar patterns are observed in article [11], in which a rise in the temperature from 1273 K to 1373 K was accompanied by a substantial intensification of sintering.

It was established that the scale of a nickel-cobalt containing precision alloy, in contrast to the unalloyed scale [11], contained in its composition additional phase of Fe_3O_4. Atoms of Ni and Co have probably partially substituted the atoms of Fe in the oxides. The presence of alloying elements predetermined a more complex character of phase transitions than that in paper [10] (Fig. 3). The resulting product of reduction consisted of γ-Fe and a certain share of residual oxides of Fe_3O_4 and FeO. FeNi formed at intermediate stages, but in the target product the prevailing amount of Ni and Co, judging by the diffractograms, was contained in the solid solution of γ-Fe. Achieving the reduction of oxide nickel- and cobalt containing components in the target product after thermal treatment at 1273 K agrees well with the results of articles [8, 9].

With a growth in the degree of reduction from 23 % to 92 %, the intensity of the Fe_3O_4, FeO and FeNi manifestation decreased while that of γ–Fe grew. A product of heat treatment with a degree of reduction of 92 % consisted of the solid solution of Co and Ni atoms in γ–Fe, as well as the remaining FeO and Fe_3O_4.

The microstructure of samples is non-uniform with the presence of particles of different size (Fig. 4). After treatment at 673 K the processes of particle sintering demonstrated a relatively low level (Fig. 4, a). The samples reduced at 1273 K had a sponge-like microstructure (Fig. 4, b), with a clearly manifested certain degree of particle sintering.

Reduction at 1573 K leads to the formation of sintered microstructure of the surface layer (Fig. 4, c). In contrast to the two previous cases, it is difficult to distinguish between separate particles – we observed an almost solid sintered layer.

Results of the phase x-ray structural studies (Fig. 3) indicate the absence of formation during reduction process of compounds and phases susceptible to sublimation. That is, there is no need to create special conditions that prevent the evaporation and loss of alloying elements with the gas phase. This also predetermines an increase in the degree of using alloying elements.

The absence of x-ray microanalysis may be considered a shortcoming of the present study. Its application would make it possible to uncover in detail the nature of phases and inclusions in the images of the microstructure.

Further development of present research may address the implementation of regulation of chemical and phase compositions of the obtained alloying additive by adding the oxides of targeted alloying elements to the charge. This would enable, on the one hand, a reduction in the expense coefficients when using a metallized product via increased alloying. On the other hand, this would open up a prospect of expanding the brand range of alloys to use the alloying additive. Difficulties that may occur when trying to develop the present study come down to the absence of a sufficient base of experimental data on the physical-chemical regularities of hydrogen reduction in the complexly-alloyed systems. This indicates the need to conduct additional research in order to identify the interaction between a complex of the alloying elements and a reducer, and to bring down their losses during thermal treatment.

Experimental-industrial testing of the metallized scale as a charge additive in the process of smelting of precision alloys of the type 29NK was carried out in the induction furnace "IST-7" at the plant PAT "Dniprospetsstal" (Ukraine). The total volume of melted alloy reached 105 t. The loading was carried out on the bottom of the furnace "under the filling". The additive was in pellets of rounded shape with density 4.7–5.3 g/cm³ of side dimensions: length and width are 60 mm, thickness is 40 mm. Temperature of the crucible of the furnace during loading (after discharge of the previous smelting) was 1450–1500 °C. The temperature of the melt during dissolution of the metallized alloying additive was 1540–1600 °C. We used the metallized scale in the amount from 25 to 90 kg/t, which allowed reduction in the consumption of metallic Ni and Co by 2.6–9.9 % of the weight of respective alloying materials. The assimilation of the alloying elements from the metallized alloying material was within a range of 94–97 %. The relatively high degree of assimilation was provided by the absence of compounds prone to sublimation and a sponge-like structure, which predetermines faster dissolution of the alloying additive in the melt of metal.

7. Conclusions

1. During kinematic research, it was established that the highest degree of hydrogen reduction of the nickel-cobalt containing precision alloy (99 %) is achieved through thermal treatment at 1273 K. This is caused by the intensification of reduction processes and a sufficient level of porosity, which ensures satisfactory gas exchange in the regions of reactions.

2. It was established that with an increase in the degree of reduction of the scale of a nickel-cobalt containing precision alloy to 92 %, basic phases underwent transformations from Fe_3O_4, Fe_2O_3, FeO to the solid solution of Co and Ni atoms in γ–Fe. The share of non-reduced oxide component in the resulting product is represented by Fe_3O_4 and FeO. We revealed the presence of FeNi at the degree of reduction from 12 % to 61 %. The microstructure of the products of scale metallization is heterogeneous, it consisted of particles with different size, level of sintering, depending on the temperature of treatment.

The resulting phases in the examined temperature range had no noticeable susceptibility to sublimation, which ensured a reduction in the losses of alloying elements when obtaining highly-alloyed metallized scale. Testing the latter during smelting of precision alloys confirmed manufacturability and efficiency of recirculating the alloying elements. At the same time, recycling of industrial wastes contributes to a reduction in the technogenic intensity of industrial regions and improves ecological safety of the environment.

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RESULTS OF EXPERIMENTAL RESEARCH INTO CORRELATIONS BETWEEN HAZARDOUS FACTORS OF IGNITION OF MATERIALS IN PREMISES

Представлені результати експериментального дослідження динаміки небезпечних факторів стану середовища в моделюючей камері, а також їх автокореляцій і парних кореляцій при різних лагах для спирту, паперу, деревини і текстилю. Встановлено, що інформативною ознакою раннього виявлення загорянь є ступінь взаємної кореляції флуктуацій факторів при нульовому лагу. Виявлено, що для розглянутих матеріалів ознакою раннього виявлення їх загоряння є коефіцієнт парної кореляції CO і температури при нульовому лагу, що має дуже високий ступінь кореляції

Ключові слова: раннє загоряння матеріалів, динаміка небезпечних факторів стану середовища, автокореляції факторів, парні кореляції факторів

Представлены результаты экспериментального исследования динамики опасных факторов состояния среды в моделирующей камере, а также их автокорреляций и парных корреляций при различных лагах для спирта, бумаги, древесины и текстиля. Установлено, что информативным признаком раннего обнаружения возгораний является степень взаимной корреляции флуктуаций факторов при нулевом лаге. Выявлено, что для рассмотренных материалов признаком раннего обнаружения их возгорания является коэффициент парной корреляции CO и температуры при нулевом лаге, обладающий высокой степенью корреляции

Ключевые слова: раннее возгорание материалов, динамика опасных факторов состояния среды, автокорреляции факторов, парные корреляции факторов

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1. Introduction

Prevention of ignition in premises is one of the most important problems of safe development of community. This causes the relevance of development of systems of fire-prevention automatic equipment. Modern systems of fire-prevention automatic equipment must enable guaranteed early detection of ignitions in premises. Recently, there have been two main trends of development of systems of fire-prevention automatic. The first trend is connected with improvement of primary sources of information on hazardous factors of early ignition in the premises based on new data processing technologies and considers actual conditions [1–3]. The issues of creation of self-adjusted primary sources of information are explored [4, 5].

The other trend is based on group processing of information on hazardous factors of early ignition and networking technologies [6]. In the framework of this trend, the authors developed new technologies of joint processing of information on two or more factors for reliable early detection of ignition in the premises. First of all, it is development of technologies of multi-sensor ignition detection [7]. A part of these technologies has received the status of EN and ISO standards [8–10]. However, because of complexity and diversity of actual ignitions, while substantiating technologies of multi-sensor detection, the properties of major hazardous factors of ignition remain insufficiently studied.

In this regard, the study of the properties of major factors of ignition of combustible materials is relevant.