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DIFFERENTIAL THERMAL ANALYSIS OF CHARGE MATERIALS FOR THE PRODUCTION OF CHROMIUM-MANGANESE LIGATURE

ДИФФЕРЕНЦИАЛЬНЫЙ ТЕРМИЧЕСКИЙ АНАЛИЗ ШИХТОВЫХ МАТЕРИАЛОВ ДЛЯ ПОЛУЧЕНИЯ ХРОМОМАРГАНЦЕВОЙ ЛИГАТУРЫ

ХРОМ-МАРГАНЕЦТІ ЛИГАТУРАНЫ АЛУ ҮШІН ШИКІҚҰРАМ МАТЕРИАЛДАРДЫҢ ДИФФЕРЕНЦИАЛДЫ ТЕРМИЯЛЫҚ ТАЛДАУЫ

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Keywords:
alloy, ligature, thermal analysis, differential scanning calorimeter, temperature.

ABSTRACT

This article investigates the physicochemical properties of initial charge materials in order to improve the efficiency of the production process of chromium-manganese ligature. The aim of the study was to determine the thermal characteristics of chromium and iron-manganese ores, as well as new silicon-aluminium alloys (ferrosilicoaluminium (FeSiAl), ferrosilicochromium (FeSiCr) dust, alumosilicomanganese (AlSiMn)).
Differential thermal analysis (DTA) was employed to study the thermal behaviour of the materials, including melting and crystallization processes, phase transitions, and chemical reactions.
The main part of the work discusses the theoretical principles of the DTA method, its applications, and provides a detailed analysis of the experimental results obtained. Particular attention was paid to the thermal properties of the initial raw materials and their changes during the process.
Thermal analysis was carried out using the STA300 "Synchronous Thermal Analyzer". The obtained data provide scientific evidence for improving the efficiency of chromium-manganese ligature production technologies.
The practical significance of the study lies in the efficient utilization of chromium and iron-manganese ores and the enhancement of reduction processes through the use of new silicon-aluminium alloys.

Түйінді сөздер:
қорытпа, лигатура, термиялық талдау, дифференциалды сканерлеуші калориметр, температура.

ТҮЙІНДЕМЕ

Бұл мақалада хром-марганецті лигатура алу үрдісінің тиімділігін арттыру мақсатында бастапқы шикізат материалдарының физика-химиялық қасиеттері зерттелді. Зерттеудің мақсаты – хром және темірлі-марганец кендері, сондай-ақ жаңа кремний-алюминийлі қорытпалардың (ферросиликоалюминий (ФСА), ферросиликохром (ФСХ) шаңы, алюмосиликомарганец (АМС)) термиялық сипаттамаларын анықтау.

Зерттеу барысында дифференциалды термиялық талдау (ДТА) әдісі қолданылды. Бұл әдіс материалдардың жылу қасиеттерін, балқу және кристалдану процестерін, фазалық ауысулар мен химиялық реакцияларды зерттеуге мүмкіндік берді.
Жұмыстың негізгі бөлімінде ДТА әдісінің теориялық негіздері, қолданылу аясы және алынған тәжірибелік нәтижелер қарастырылды. Әсіресе, бастапқы шикізат материалдарының термиялық қасиеттерін және олардың үрдіс кезіндегі өзгерістерін анықтау мақсатында жүргізілген талдауларға ерекше назар аударылды.
Термиялық талдау STA300 маркалы «Synchronous Thermal Analyzer» құрылғысында жүргізілді. Алынған нәтижелер хром-марганецті лигатураны алу технологиясында қолданылатын процестердің тиімділігін арттыруға арналған ғылыми деректерді ұсынды.
Зерттеудің практикалық маңызы – хром және темірлі-марганец кендерін тиімді пайдалану, сондай-ақ жаңа кремний-алюминийлі қорытпаларды қолдану арқылы тотықсыздандыру үрдісін жетілдіру болып табылады.

Ключевые слова:
сплав, лигатура, термический анализ, дифференциальный сканирующий калориметр, температура.

АННОТАЦИЯ
В данной статье исследованы физико-химические свойства исходных шихтовых материалов с целью повышения эффективности процесса получения хромомарганцевой лигатуры. Цель исследования заключалась в определении термических характеристик хромовых и железо-марганцевых руд, а также новых кремний-алюминиевых сплавов (ферросиликоалюминий (ФСА), пыль ферросиликохрома (ФСХ), алюмосиликомарганец (АМС)).
Для проведения анализа применялся метод дифференциального термического анализа (ДТА), который позволяет изучать тепловые свойства материалов, процессы плавления и кристаллизации, фазовые превращения и химические реакции.
В основной части работы рассмотрены теоретические основы метода ДТА, области его применения, а также подробно проанализированы полученные экспериментальные результаты. Особое внимание уделено изучению термических свойств исходных материалов и их изменений в ходе процесса.
Термический анализ проводился на приборе STA300 «Synchronous Thermal Analyzer». Полученные данные предоставляют научное обоснование для повышения эффективности технологий получения хромомарганцевой лигатуры.
Практическая значимость исследования заключается в более рациональном использовании хромовых и железо-марганцевых руд, а также в улучшении восстановительных процессов с применением новых кремний-алюминиевых сплавов.

INTRODUCTION

In modern metallurgy, the study of the thermal properties of charge materials plays a key role in the development of new alloys and ligatures. DTA is one of the main methods for studying the physico-chemical properties of materials (Sitnikova, Ponomarea & Uspenskaya, 2021). Using DTA, it is possible to determine such characteristics as melting points, decomposition, and phase transformations, which makes it possible to effectively control the temperature conditions of production processes. This method consists of tracking the temperature difference between the sample and the reference material as a function of time or temperature, while the temperature of the sample changes according to a preset program in a specific atmosphere (Makhambetov et. al., 2022; Myngzhassar, 2025).

When obtaining ligatures, the study of the interaction of charge components and their thermodynamic behavior is of particular importance (Zhuniskaliyev et. al., 2024). This makes it possible to optimize the composition of the charge, improve the quality of the obtained ligatures and increase the efficiency of metallurgical processes.

Differential thermal analysis allows precise determination of phase changes, chemical reactions, melting and crystallization processes of materials (Ido, Iwamoto & Kondoh, 2020). These studies provide a deeper understanding of the properties of the raw materials needed to produce chromium-manganese ligature, and ensure the correct choice of its composition (Wang et. al., 2021). The data obtained helps predict the behavior of materials in various temperature conditions, which minimizes production costs and increases the efficiency of technological operations.

MATERIALS AND METHODS

The research work assumes the use of domestic chromium and manganese ores as raw materials for the development of chromium-manganese ligature technology, as well as new silicon-aluminum alloys (ferrosilicon aluminum, ferrosilicon chromium dust, aluminosilicon manganese) as reducing agents.

Their thermodynamic properties, including melting points, phase transformations, and reduction reactions, are studied in the preparation of charge materials such as FeSiCr dust, FeSiAl, AlSiMn, and chromium and iron-manganese ores. FeSiCr dust rich in silicon and chromium demonstrates important characteristics for optimizing metallurgical processes such as reduction and melting. FeSiAl with a high content of aluminum and silicon is actively used as a reducing agent, and its thermal behavior also plays a key role in the steel alloying process (Nurumgaliyev et. al., 2023; Mukhambetgaliyev et. al., 2021). AlSiMn containing manganese, silicon and aluminum affects the properties of the charge, improving the quality of the obtained alloys (Shabanov, 2016; Yessengaliev, 2020). Cr and Mn ore are the main sources for obtaining chromium-manganese ligatures, which are necessary for the production of ferroalloys. DTA helps to accurately determine their behavior at high temperatures, which is important for the development of efficient metallurgical technologies (Cheng et. al., 2019; Daver et. al., 2016).

To study the physico-chemical properties of these raw materials, namely, to perform differential thermal analysis, the device «Synchronous Thermal Analyzer» brand STA300 was used (**Figure 1).** This device allows you to determine phase changes, reactivity, and changes that occur when temperature affects the composition of raw materials.

Figure 1. «Synchronous Thermal Analyzer» STA300

Note – compiled by the authors

The composition and characteristics of this equipment are as follows: a differential scanning calorimeter (DSC) is used to measure parameters such as melting, crystallization, phase transitions, reaction temperature, heat of reaction, heat of combustion, and specific heat (Aliberti et. al., 2025). These data allow us to accurately determine the thermodynamic behavior of materials and their reactions to temperature changes. In addition, thermal stability, decomposition, oxidation and reduction, adsorption and desorption properties, as well as the content of free and crystalline water in materials are investigated using thermogravimetric analysis (TGA) (Almyashev et. al., 2017; Lotov et. al., 2022). The device is also used to calculate the ratios of compounds, which contributes to a more accurate determination of their chemical composition and reaction characteristics at high temperatures.

The furnace body is heated by a double-row winding made of precious metal alloy wire, which reduces interference, as well as resists high temperature and oxidation. The dish sensor has a wide range of tests, high temperature resistance and resistance to oxidation and corrosion, which ensures reliable operation of the device in difficult conditions. The weighing system uses imported components, which are characterized by high stability and good repeatability of measurements. The furnace body has double-layer insulation, which minimizes heat loss and increases the energy efficiency of the device. The heating of the device is controlled using a proportional-integral-differential (PID) controller, which ensures the accuracy of temperature control and minimal pulse fluctuations, ensuring stable operation of the device during operation.

The temperature range of the device is from room temperature to 1550°C with a resolution of 0.01°C and fluctuations up to 0.1°C. The heating rate is from 0.1 to 100 °C/min, with temperature control according to the PID algorithm. The temperature maintenance time is up to 300 minutes. The weight measurement range is from 0.01 mg to 3 g (expandable to 30 g). The DSC range is from 0 to 600 MW with a resolution of 0.001 MW. The atmosphere of the device is inert, oxidizing or reducing.

RESULTS AND DISCUSSION

This section presents the differential thermal analysis results (**Figures 2–6**) of chromium and iron-manganese ores, as well as new silicon–aluminum alloys. The obtained thermograms reveal endothermic and exothermic effects, melting and crystallization processes, and phase transformations. A comparative discussion highlights the differences in thermal behaviour of natural ores and synthetic alloys, providing insights into their reactivity and stability under high-temperature conditions relevant to chromium–manganese ligature production.

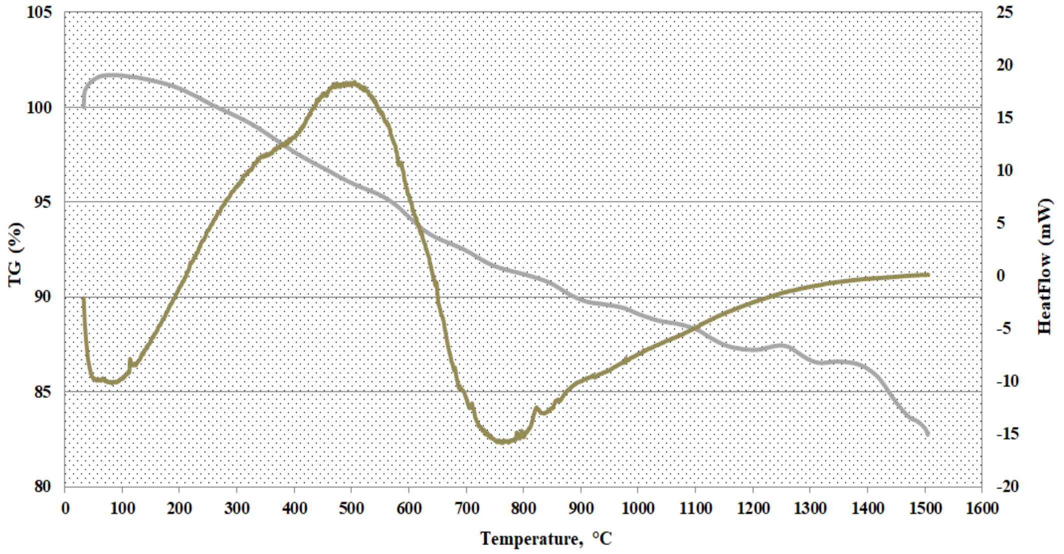

Figure 2. Chromium ore derivatogram.
Note – compiled by the authors

Figure 2, which is based on the derivatogram of chromium ore, clearly illustrates a sequence of thermal effects that characterize the behavior of the ore upon heating. These effects reflect the complex physicochemical transformations that occur within the material and determine its suitability for further metallurgical processing.

In the initial stage of heating, within the temperature range of 100–400 °C, the first endothermic peak is recorded. This effect is mainly attributed to the removal of physically adsorbed water and volatile components trapped in the pore structure of the ore. Such dehydration processes are typical for natural ores and indicate the release of surface-bound moisture, which does not alter the chemical composition but affects the overall stability of the material during subsequent heating.

As the temperature rises to the range of 500–750 °C, a distinct exothermic peak is observed. This thermal effect is linked to oxidation processes, where chromium-bearing minerals undergo structural rearrangements accompanied by the release of heat. At this stage, chromium compounds tend to oxidize, forming various intermediate oxides. The appearance of such a pronounced exothermic effect highlights the high reactivity of chromium phases and marks the onset of important chemical transformations that influence the reduction behavior of the ore in later stages.

Further heating to 800–1400 °C reveals a series of phase transformations. In this temperature interval, stable chromium oxides such as Cr_2O_3 are formed, which represent the thermodynamically favorable phases under oxidizing conditions. Additionally, in the presence of impurities (iron, manganese, or silica), solid-state interactions may take place, leading to the formation of mixed oxides or spinel-type compounds. These transformations are of particular significance for ferroalloy production, as they determine the reducibility of chromium and its potential recovery in the smelting process.

Beyond 1400 °C, the derivatogram curve becomes relatively stable, with no major thermal effects recorded. The mass of the sample remains almost unchanged, which indicates that most dehydration, oxidation, and phase transformation processes have been completed by this point. The stabilization of the system at such high temperatures suggests that chromium ore reaches a thermally resistant state, with only minor structural adjustments occurring.

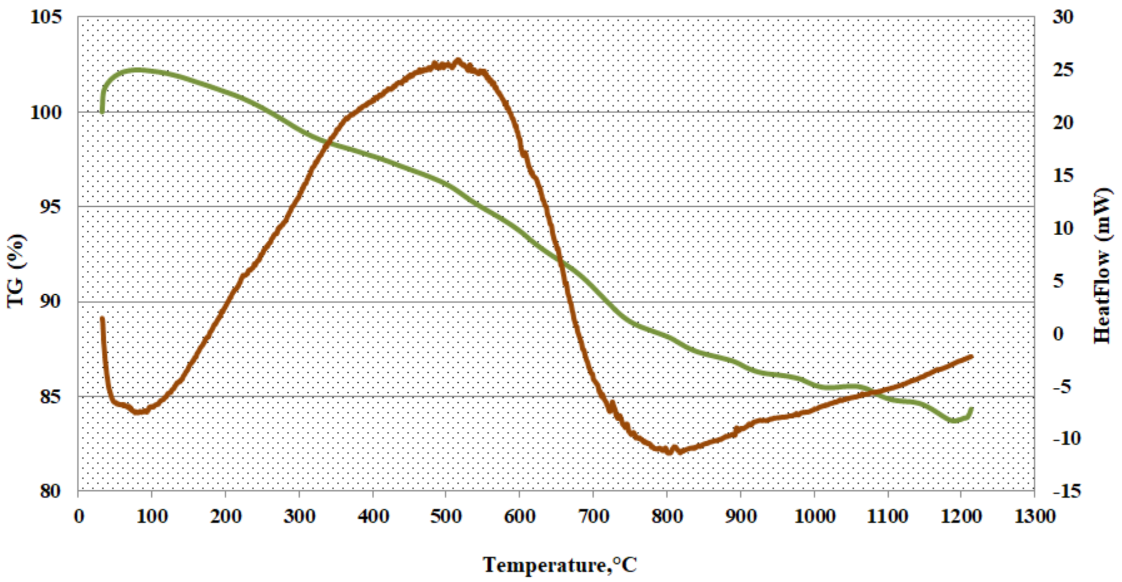

Figure 3. Iron-manganese ore derivatogram

Note – compiled by the authors

Figure 3 presents the derivatogram of iron-manganese ore, which reveals a sequence of characteristic thermal effects during heating. These effects reflect the multistage physicochemical processes that occur within the ore and provide valuable information about its thermal stability and reactivity.

In the initial heating stage (100–300 °C), an endothermic effect is recorded, which corresponds to the removal of physically adsorbed water and volatile substances. This dehydration process is typical for natural ores and indicates the release of weakly bound components without significant changes in the mineralogical structure. Such processes are important because they affect the porosity and surface area of the ore, which in turn influence its reactivity in subsequent high-temperature stages.

In the range of 350–750 °C, a distinct exothermic effect is observed. This is attributed to the oxidation of manganese-bearing phases and the formation of manganese oxides such as MnO and Mn_2O_3. The release of heat during this process reflects the high reactivity of manganese minerals in this temperature region. The formation of intermediate oxides is a critical stage, as it determines the oxidation state of manganese and its subsequent reducibility in metallurgical processes.

Further heating between 750–1200 °C leads to phase transformations and solid-state interactions among the ore components. At this stage, structural rearrangements occur, including the stabilization of manganese oxides and their possible interaction with associated impurities such as iron, silica, or alumina. The formation of complex oxide phases or spinel-type compounds can also take place, which has a direct impact on the melting behavior and reduction characteristics of the ore under smelting conditions.

Above 1200 °C, the derivatogram becomes relatively stable, indicating that the majority of thermal reactions are completed. The system reaches a state of thermal resistance, with only minor changes in mass or heat flow. This stabilization suggests that manganese ore achieves structural equilibrium at elevated temperatures.

Overall, the thermal analysis of iron-manganese ore provides detailed insight into its behavior under heating. The sequence of dehydration, oxidation, and high-temperature phase transformations is crucial for understanding its reduction mechanisms and optimizing its use in chromium–manganese ligature production.

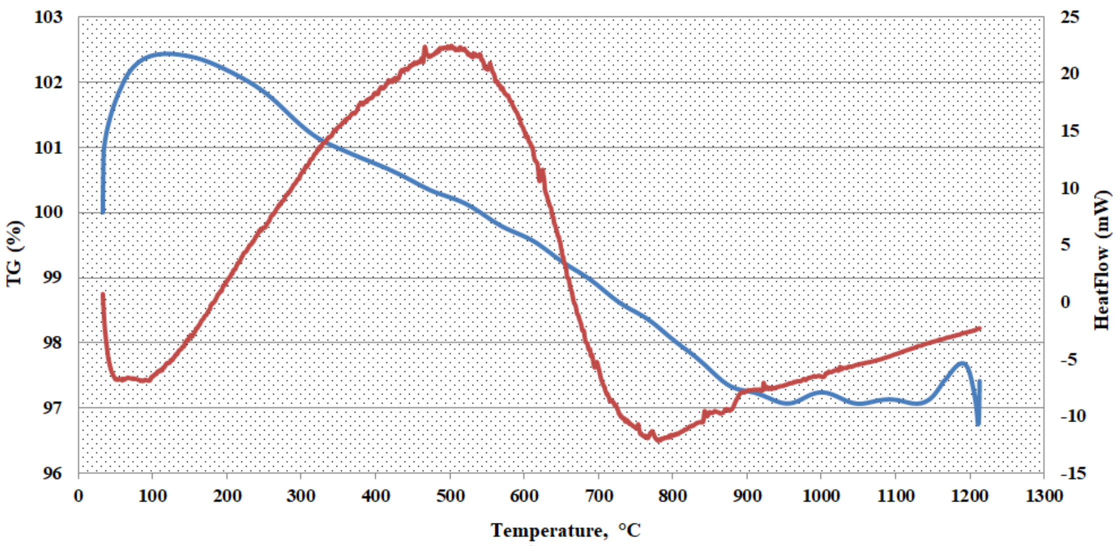

Figure 4. FeSiAl derivatogram.

Note – compiled by the authors

The derivatogram of FeSiAl (Figure 4) demonstrates several distinct thermal events that characterize the material's behavior upon heating. At temperatures below 200 °C, a minor endothermic effect is recorded, which can be attributed to the release of adsorbed water and volatile surface impurities. As the temperature increases to 400–670 °C, a more pronounced endothermic reaction takes place, likely related to the decomposition of unstable compounds or partial rearrangement within the alloy structure. Between 670 and 850 °C, noticeable phase transitions occur, reflecting changes in the solid-state organization and the possible emergence of new phases. At higher temperatures (850–1200 °C), melting phenomena and the stabilization of thermodynamically favorable phases are observed, accompanied by a distinct shift in the heat flow curve. These features emphasize the complex structural nature of ferrosilicoaluminium and its transformation under high-temperature conditions.

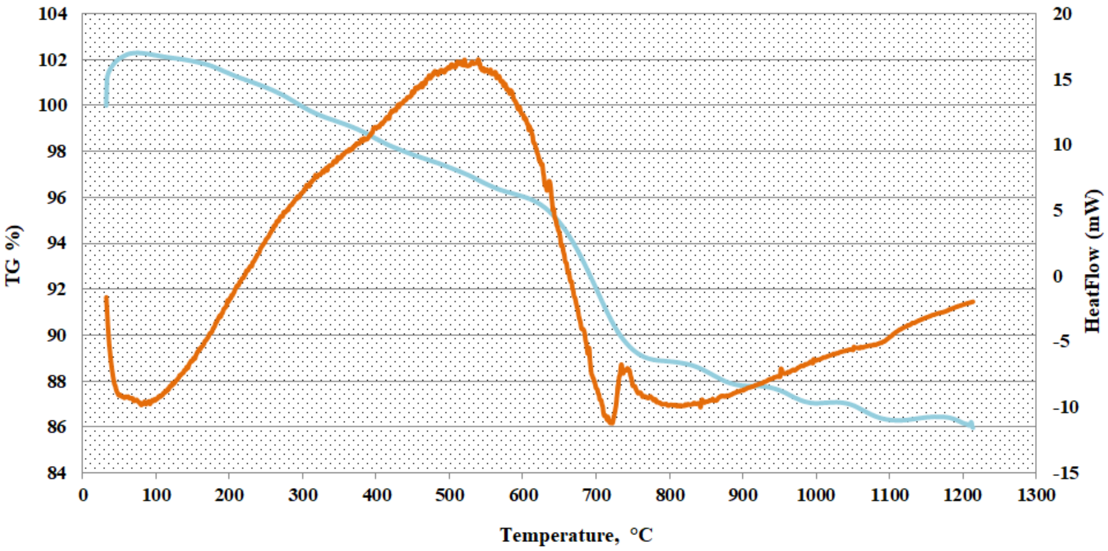

Figure 5. FeSiCr dust derivatogram.

Note – compiled by the authors

The FeSiCr dust derivatogram (Figure 5) illustrates a sequence of thermal effects that describe the material's transformations during heating. In the region up to 200 °C, a weak endothermic peak is observed, caused by the removal of physically adsorbed water and light volatile compounds. Between 200 and 660 °C, a smooth decrease in mass takes place, which can be explained by the decomposition of unstable surface compounds, such as thin oxide films or residual contaminants.

Further heating to 660–850 °C initiates structural modifications within the dust particles. At this stage, new phases may appear, while existing ones undergo rearrangement, which is reflected by a slight endothermic response on the DSC curve. Finally, in the high-temperature interval of 850–1200 °C, more complex processes are detected, including the onset of melting in certain components and the formation of stable crystalline phases. These transformations are accompanied by noticeable changes in heat flow, reflecting the transition of the material into thermodynamically stable states.

Overall, the obtained data provide important information about the thermal behavior of FeSiCr dust, confirming its complex multistage transformations. Such results are essential for understanding the mechanisms of phase changes during high-temperature metallurgical processes, as well as for assessing its role as a reducing agent in the production of chromium-manganese alloys.

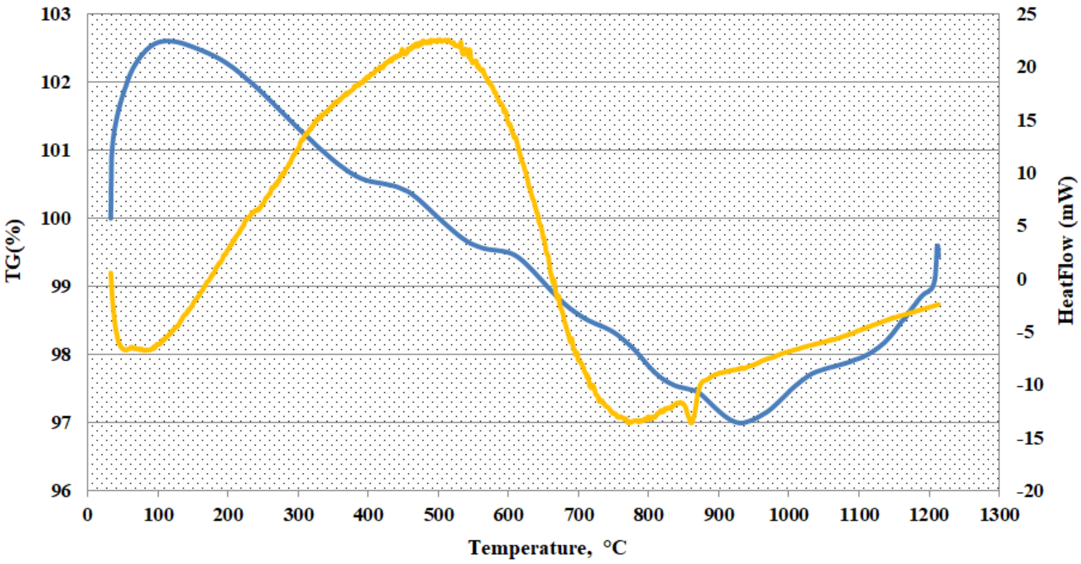

Figure 6. AlSiMn derivatogram.

Note – compiled by the authors

The derivatogram of AlSiMn (Figure 6) demonstrates a series of characteristic thermal events that reflect the behavior of the alloy during heating. At temperatures up to 200 °C, a weak endothermic effect is recorded, which corresponds to the removal of adsorbed water and light volatile components. In the range of 400–700 °C, a more pronounced endothermic process is observed, most likely related to the decomposition of unstable intermetallic phases or restructuring within the alloy's microstructure.

As the temperature rises to 700–850 °C, an exothermic effect becomes evident. This stage is indicative of redox reactions and the emergence of secondary phases, which mark the material's transition to a more complex structural state. At higher temperatures, between 850 and 1200 °C, significant phase transformations occur. These processes are typical for aluminum-based alloys of the AlSiMn system and may include crystallization phenomena or the stabilization of new intermetallic phases. The changes are accompanied by a smooth shift in heat flow, reflecting the gradual adaptation of the alloy to high-temperature conditions.

CONCLUSION

The present study focused on the thermal analysis of charge materials used in the production of chromium–manganese ligatures, including FeSiCr dust, FeSiAl, AlSiMn, as well as chromium and iron-manganese ores. DTA made it possible to identify the key thermal properties of these materials, such as characteristic melting points, phase transformations, and reduction reactions. These findings provide a detailed understanding of the mechanisms that govern the behavior of the raw materials under high-temperature conditions.

The results indicate that chromium and iron-manganese ores, despite being low-grade, demonstrate predictable and consistent thermal behavior, making them suitable for further metallurgical processing. At the same time, the use of complex silicon–aluminum alloys such as FeSiCr dust, FeSiAl, and AlSiMn as reducing agents has shown significant potential due to their favorable thermodynamic properties and ability to participate effectively in reduction reactions. This confirms their value in the optimization of the reduction process and the stabilization of phase composition during alloy production.

A comparative assessment of the investigated materials highlights the advantages of combining ores with silicon–aluminum alloys, which ensures efficient reduction, improved phase transformations, and the formation of stable products at high temperatures. Such results underline the possibility of designing more rational charge compositions aimed at obtaining high-quality chromium–manganese ligatures.

From a practical perspective, the outcomes of this study provide a strong basis for improving existing metallurgical processes. The incorporation of the studied materials can contribute to higher productivity, lower energy consumption, and improved quality of the final product. Furthermore, the findings support the implementation of innovative technological approaches in ferroalloy production, paving the way for the efficient utilization of low-grade ores and secondary raw materials.

In summary, this research not only contributes to the theoretical understanding of the thermal behavior of ores and alloys but also offers practical recommendations for the development of advanced metallurgical technologies. The obtained results have both scientific and industrial relevance, emphasizing their importance for the sustainable growth and modernization of the ferroalloy industry.

CONFLICT OF INTEREST: The authors declare no conflict of interest.

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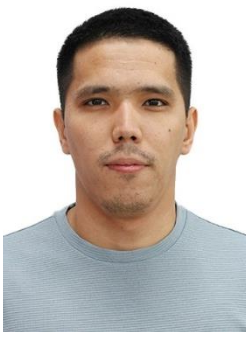

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