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Оригинальная статья

EFFECT OF MECHANICAL PROCESSING ON REDUCTION OF IRON OXIDES IN MAN-MADE RAW MATERIALS

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Abstract. The study considers ways to increase the efficiency of reduction of iron oxides from man-made waste (dust from electric arc furnaces) using mechanochemical activation (MCA), grinding and pressing. The analysis of chemical and phase compositions of the dust samples was carried out, which made it possible to identify their potential for processing. The experiments included a study of the effect of grinding and pressing at pressures up to 300 MPa on the materials' phase composition, as well as an assessment of the effects of coke addition during MCA. To study the effect of pressing pressure on the reduction processes, briquettes were fired at a temperature of 1200 °C. The results showed that the degree of iron metallization increases with an increase in pressing pressure: concentration of metallic iron reaches 19 % at a pressure of 300 MPa, which is higher compared to 17 % in the initial state without pressing. The novelty of the work lies in optimizing the pressing parameters and demonstrating its effect on the iron reduction process. The proposed conditions make it possible to increase the efficiency of processing man-made waste, which can be used to improve the environmental and economic components of production.

Keywords: mechanochemical activation, steelmaking dust, metallization, oxide reduction, zinc extraction, phase composition, secondary resources, waste recycling

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ВОЗДЕЙСТВИЕ МЕХАНИЧЕСКОЙ ОБРАБОТКИ НА ПРОЦЕССЫ ВОССТАНОВЛЕНИЯ ОКСИДОВ ЖЕЛЕЗА В ТЕХНОГЕННОМ СЫРЬЕ

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Аннотация. Рассмотрены способы повышения эффективности восстановления оксидов железа из техногенных отходов (пылей дуговой сталеплавильной печи) с применением механохимической активации, помола и прессования. Проведен анализ химического и фазового составов образцов пылей, что позволило выявить их потенциал для переработки. Эксперименты включали исследование влияния помола и прессования при давлениях до 300 МПа на фазовый состав материалов, а также оценку эффекта добавления кокса в процессе механохимической активации. Для изучения влияния давления прессования на восстановительные процессы был проведен обжиг брикетов при температуре 1200 °C. Полученные результаты показали, что степень металлизации железа возрастает при увеличении давления пресс-

сования: содержание металлического железа достигает 19 % при давлении 300 МПа, что выше по сравнению с 17 % в исходном состоянии без прессования. Новизна работы заключается в оптимизации параметров прессования и демонстрации его влияния на процесс восстановления железа. Предложенные условия позволяют повысить эффективность переработки техногенных отходов, что может быть использовано для улучшения экологической и экономической составляющих производства.

Ключевые слова: механохимическая активация, сталеплавильные пыли, металлизация, восстановление оксидов, извлечение цинка, фазовый состав, вторичные ресурсы, переработка отходов

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INTRODUCTION

The processing of man-made raw materials has become one of the key challenges in modern industry and environmental management. Man-made raw materials include various wastes and by-products generated during industrial processes. These materials often contain valuable components, such as metals, minerals, and chemical compounds, which can be extracted and reused. The efficiency of raw material processing can be enhanced through grinding and pressing, i.e., mechanochemical activation (MCA) [1; 2].

Mechanochemical activation is a process in which mechanical action is applied to solid substances, leading to changes in their physicochemical properties. This action can involve operations such as grinding, pressing, rolling, or other forms of mechanical impact. The MCA process is employed to enhance material reactivity [3], modify phase composition [4], improve interactions between components, and activate chemical reactions [5] that would otherwise occur slowly or not at all under standard conditions.

Outlined below are the key aspects of MCA.

- **Grinding and lattice destruction.** During grinding, the crystalline lattice of solid substances is disrupted, leading to the formation of defects and an increase in the specific surface area. This enhances the material's reactivity, as defects can serve as nucleation centers for new phases and initiate chemical reactions [6 – 8].

- **Formation of active centers.** Mechanical impact generates active centers on the particle surfaces, including free radicals, lattice defects, and surface irregularities. These active centers can trigger chemical reactions that would otherwise occur very slowly or require high temperatures and catalysts under normal conditions [9; 10].

- **Phase composition changes.** Mechanochemical activation can significantly alter a material's phase composition. For example, new phases that were absent in the initial material may form, or existing phases may transform into more stable or reactive forms [11 – 13].

- **Increased chemical activity.** Mechanochemically activated materials often demonstrate heightened chemical activity. This enhanced reactivity can be leveraged

to accelerate the reduction of metals from oxides, synthesize new compounds, or break down otherwise stable chemical bonds [14 – 16].

- **Lower reaction temperatures.** Mechanochemical activation enables many chemical reactions to occur at lower temperatures than would otherwise be necessary. This effect is attributed to the accumulation of mechanical energy within the material, which helps to overcome the reaction's energy barrier [17; 18].

Thus, MCA is an important tool for controlling the physicochemical properties of materials, enabling the development of innovative technologies and processes.

In earlier experiments on the conditions of the pyrometallurgical reduction of oxide scale, it was found that increasing the pressing pressure of the scale during its preparation for firing from 0 to 300 MPa doubled its metallization degree during heating, while the onset temperature of metallization decreased by more than 40 °C [19]. It was hypothesized that the observed effects during the pyrometallurgical reduction of oxide scale result from the mechanochemical activation of iron oxides in the scale during pressing. Accordingly, the objective of this study is to confirm this hypothesis, optimize the pressing parameters, and demonstrate the impact of mechanical processing of raw materials on the iron reduction process.

EVALUATION OF FRANKLINITE DECOMPOSITION

POTENTIAL DURING MCA

The effects of grinding and pressing pressure on the phase composition of electric arc furnace (EAF) dust were studied. To evaluate the influence of MCA on the phase composition of EAF dust, the tested dust samples were mixed to prepare an averaged sample, which was then ground for 2 min and pressed at pressures ranging from 0 to 300 MPa. The composition of the raw mixture in the first series and the processing conditions are presented in Table 1.

In the second series, coke was added to the dust, and the raw mixture was subjected to MCA. The composition of the raw mixture in the second series and the processing conditions are shown in Table 2.

Table 1. Composition of raw material mixture of the first series and processing mode**Таблица 1. Состав сырьевой смеси первой серии и режимы обработки**

Sample ID	Composition				Grinding, min	Pressing pressure, MPa
	EAF dust		coke			
	%	g	%	g		
1.1	100	20	0	0	0	0
1.2					2	0
1.3					2	100
1.4					2	200
1.5					2	300

Таблица 2. Состав сырьевой смеси второй серии и режимы обработки**Table 2. Composition of raw material mixture of the second series and processing mode**

Sample ID	Composition				Grinding, min	Pressing pressure, MPa
	EAF dust		coke			
	%	g	%	g		
2.1	80	16	20	4	0	0
2.2					2	0
2.3					2	100
2.4					2	200
2.5					2	300

The processed products were subjected to quantitative phase analysis.

Quantitative *X*-ray phase analysis was carried out using a STADI-P diffractometer (STOE, Germany). Data were collected with CuK_α radiation (40 kV, 30 mA), a graphite monochromator, within a scattering angle range of $2\theta = 10 \div 70^\circ$, with a step size of 0.02° and a dwell time of 2 s per step. The results were analyzed using the PDF-2 database (Release 2008 RDB 2.0804).

ASSESSMENT OF MCA'S IMPACT ON THE PHASE

COMPOSITION OF EAF DUST

The phase analysis results for samples 1.1 – 1.5 without coke are presented in Fig. 1.

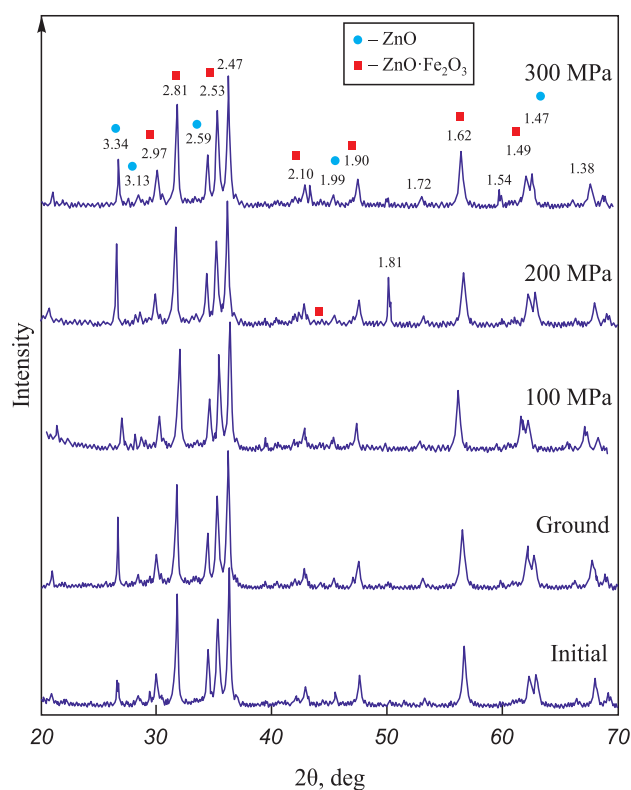
Analysis of the ground and pressed samples reveals that the intensity of the *X*-ray spectrum varies cyclically with changes in pressing pressure. Table 3 and Fig. 1 illustrate the variations in the phase composition of the samples under different processing conditions.

The findings indicate an inverse relationship in compound content. As the pressing pressure increases to 150 MPa, the ZnO content in the sample rises, while

the franklinite ($\text{ZnO} \cdot \text{Fe}_2\text{O}_3$) content decreases. Further increasing the pressing pressure to 300 MPa leads to a rise in franklinite ($\text{ZnO} \cdot \text{Fe}_2\text{O}_3$) content and a corresponding decrease in ZnO content. Therefore, it is crucial to monitor and maintain an optimal pressing pressure to achieve the desired compound composition in the final product.

Phase analysis results for samples 2.1 – 2.5 with coke are presented in Fig. 2.

Analysis of the phase composition of the ground and pressed samples indicates that, similar to the samples without coke, the intensity of the entire *X*-ray spectrum

**Fig. 1.** Results of phase analysis of the samples 1.1 – 1.5**Рис. 1.** Результаты фазового анализа проб 1.1 – 1.5**Table 3. Phase composition in the samples depending on processing modes****Таблица 3. Содержания фаз в пробах в зависимости от режимов обработки**

Sample ID	Pressing pressure, MPa	Phase composition, wt. %	
		ZnO	$\text{ZnO} \cdot \text{Fe}_2\text{O}_3$
1.1	0	34.9	44.6
1.2	50*	36.1	43.0
1.3	100	42.6	37.3
1.4	200	38.0	39.1
1.5	300	34.9	44.5
* – grinding designation.			

changes cyclically depending on the pressing pressure. Table 4 and Fig. 2 present the variations in phase composition of the samples under different processing conditions.

The test results indicate that as the pressing pressure increases, the content of free ZnO initially decreases and then sharply rises. In the initial sample, the phase ratio of ZnO/ZnO·Fe₂O₃ is 37.4/40.7, whereas after complete MCA, this ratio shifts to 46.6/31.6. This change in phase quantities is likely due to interaction with coke according to the reaction

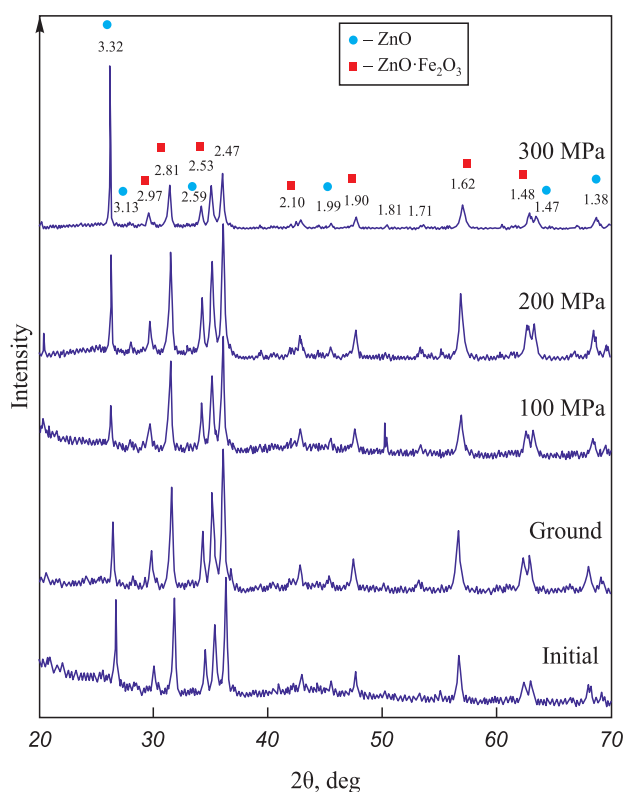


Fig. 2. Results of phase analysis of the samples 2.1 – 2.5

Рис. 2. Результаты фазового анализа проб 2.1 – 2.5

Table 4. Phase composition in the samples 2.1 – 2.5 depending on processing modes

Таблица 4. Содержания фаз в пробах 2.1 – 2.5 в зависимости от режимов обработки

Sample ID	Pressing pressure, MPa	Phase composition, wt. %	
		ZnO	ZnO·Fe ₂ O ₃
2.1	0	37.4	40.7
2.2	50*	34.1	43.3
2.3	100	32.1	43.4
2.4	200	35.6	41.5
2.5	300	46.6	31.6

* – grinding designation.

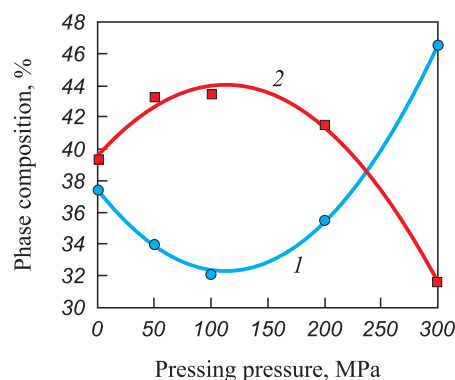


Fig. 3. Phase composition in the samples 2.1 – 2.5 depending on processing modes:

1 – ZnO; 2 – ZnO·Fe₂O₃

Рис. 3. Содержания фаз в пробах 2.1 – 2.5 в зависимости от режимов обработки:

1 – ZnO; 2 – ZnO·Fe₂O₃

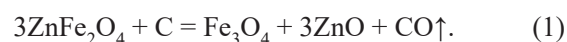


Fig. 3 shows the dependence of ZnO and ZnO·Fe₂O₃ phase content on pressing pressure.

FIRING OF PRESSED SAMPLES

To evaluate the effect of pressing pressure on the phase composition of fired products, a raw mixture was prepared using electric arc furnace (EAF) dust, coke, and a dry binder component with a content of 10 %. The components of the raw mixture were co-ground. After grinding, a liquid binder component was added to the raw mixture, which was then briquetted under pressures of 0, 100, 200 and 300 MPa. The composition of the binder is provided in the patent [20], while the composition of the raw mixture for firing and its processing conditions are listed in Table 5.

Before briquetting, a binder was introduced, consisting of ladle furnace slag (LFS), liquid glass, and hydrofluorosilicic acid (HFSA). The LFS contains approximately 40 % dicalcium silicate (2CaOSiO₂), which reacts with liquid glass, causing it to harden about 30 min after mixing and forming water-resistant tobermorite-like calcium-sodium hydrosilicates. This time is sufficient for briquetting to be carried out. Once briquetting and complete hardening are achieved, the briquettes gain high strength. Hydrofluorosilicic acid also reacts with liquid glass, promoting its hardening [21]. Additionally, the acid acts as a fluxing additive. It reacts with calcium oxide in the slag to form fluorite (fluorspar), which is a strong flux.

In [22], it was shown that when the binder content is less than 10 %, a non-diffusion reduction mode of iron oxides is implemented. In this mode, the degree of metalization is highly dependent on pressing pressure. When the binder content reaches 10 %, a liquid phase appears,

Table 5. Composition of the raw mixture for firing and its processing mode

Таблица 5. Состав сырьевой смеси для обжига и режимы ее обработки

Sample ID	Composition										Pressing pressure, MPa
	EAF dust		LFS slag		coke, (above 100 %)		Liquid glass-3.0 γ-1.2		HFSA γ-1.08		
	%	g	%	g	%	g	%	mL	%	mL	
3.1	90	18	10	2	20.0	4	7.5	1.5	3.75	0.75	0
3.2	90	18	10	2	20.0	4	7.5	1.5	3.75	0.75	100
3.3	90	18	10	2	20.0	4	7.5	1.5	3.75	0.75	200
3.4	90	18	10	2	20.0	4	7.5	1.5	3.75	0.75	300

and a diffusion reduction mode of iron oxides occurs. In this mode, the degree of metallization does not depend on pressing pressure and remains approximately the same across the entire pressure range.

The coke content corresponds to the stoichiometry of iron oxides and carbon, plus an additional 15 % to account for the ash content of the coke.

Dry briquettes were fired at temperatures up to 1200 °C for 1 h. The firing temperature matched the conditions for completing the metallization process [22]. An isothermal holding period of 30 min was maintained at 1200 °C. The overall appearance of the fired samples is shown in Fig. 4.

The fired samples in Fig. 4 clearly show droplets of metallic iron.

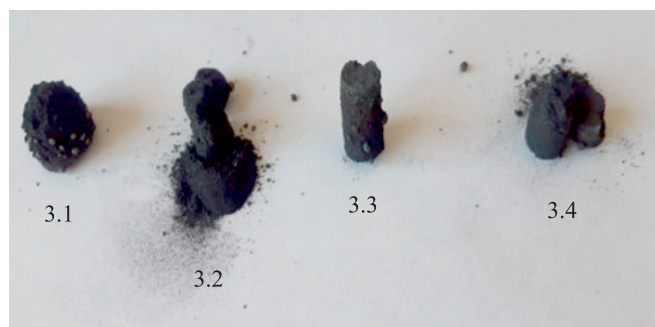


Fig. 4. General view of the fired samples

Рис. 4. Общий вид обожженных образцов

Table 6. Phase composition in the studied samples

Таблица 6. Содержание фаз в исследованных пробах

Sample ID	Phase composition, wt. %	
	Fe _{met}	2CaO·SiO ₂ , 3CaO·SiO ₂
3.1	17.1	82.9
3.2	16.3	83.7
3.3	17.4	82.6
3.4	19.0	80.8

The firing products were subjected to phase analysis, and the phase content of the studied samples is presented in Table 6.

The test results indicate that as the pressing pressure increases, the metallic iron content initially decreases but then rises, reaching 19 % at 300 MPa compared to 17 % in the initial state without pressing. Based on these findings, it is recommended to maintain a pressing pressure of 300 MPa, as lower pressures may negatively impact the degree of metallization.

RESULTS AND DISCUSSION

The research demonstrated a significant influence of pressing pressure on the phase composition and reduction processes of iron oxides in ASF dust. In the first series of experiments, conducted on samples without coke addition, cyclic variations in phase content were observed depending on the pressing pressure (Table 3). At pressures up to 150 MPa, the ZnO content increased, while the content of franklinite ($\text{ZnO} \cdot \text{Fe}_2\text{O}_3$) decreased. However, at higher pressures up to 300 MPa, the opposite effect was noted: ZnO content decreased, and franklinite content increased. These findings highlight the necessity of controlling pressing pressure to achieve the desired phase ratio in the final product.

In the second series of experiments, involving samples with coke addition (Table 4), phase analysis results similarly demonstrated cyclic variations in the contents of ZnO and $\text{ZnO} \cdot \text{Fe}_2\text{O}_3$ depending on pressing pressure. A substantial increase in free ZnO content at 300 MPa indicates the occurrence of the reduction reaction of franklinite (ZnFe_2O_4) involving carbon. The interaction, described by Equation (1), results in the formation of magnetite (Fe_3O_4), zinc oxide (ZnO), and carbon monoxide (CO), confirming the role of MCA in the breakdown of franklinite and the reduction of iron oxides.

The results of evaluating the effect of pressing pressure on firing processes (Table 6) showed that as pressure increased from 0 to 300 MPa, the metallic iron content initially decreased but subsequently increased, reaching

a maximum of 19 % at 300 MPa. These findings indicate that the optimal pressing pressure for maximizing iron metallization is 300 MPa. Lower pressures may adversely affect the reduction process, reducing the proportion of metallic iron in the final product.

Thus, the study confirms that MCA occurring during the pressing of EAF dust enhances the reduction processes of iron oxides. Optimizing pressing parameters, particularly maintaining a pressure of 300 MPa, ensures the highest metallization efficiency. This optimization offers promising opportunities to improve the productivity and environmental performance of pyrometallurgical waste processing.

CONCLUSIONS

It has been demonstrated that MCA significantly influences the phase composition of EAF dust, both with and without coke addition. In samples without coke, the phase composition changes cyclically with variations in pressing pressure. At pressures up to 150 MPa, the zinc oxide (ZnO) content increases, while the franklinite ($\text{ZnO} \cdot \text{Fe}_2\text{O}_3$) content decreases. However, with further pressure increases to 300 MPa, the franklinite content rises, and the free ZnO content decreases, indicating the need for precise pressure control to achieve the desired phase composition.

In samples with coke addition, a similar cyclic change in phase composition is observed depending on the pressing pressure. As the pressure increases, the free ZnO content initially decreases but then sharply rises. These changes are likely attributed to the reaction between franklinite and coke, resulting in the formation of magnetite (Fe_3O_4), ZnO, and carbon monoxide.

Firing of pressed samples revealed that as the pressing pressure increases, the metallic iron content initially decreases but subsequently rises, reaching a maximum at 300 MPa. This suggests that a pressing pressure of 300 MPa is optimal for achieving a high degree of metallization, while lower pressures may negatively affect the quality of the final product.

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Вклад авторов

M. V. Kleonovskii – development of the research concept and tasks, conducting the experiments, grinding and pressing of the studied samples at various pressures, analysis of the chemical and phase composition of EAF dusts and their changes during mechanochemical activation.

O. Yu. Sheshukov – scientific guidance, development of the methods for samples briquetting, firing them with subsequent analysis of the phase composition.

M. A. Mikheenkov – analysis of the effect of mechanochemical activation on the phase composition of EAF dust, study of the effect of grinding and pressing pressure on reduction of iron oxides, interpretation of the data obtained as a result of X-ray phase analysis of samples, study of interaction of the raw mixture components.

A. M. Mikheenkov – theoretical substantiation of the study, literary analysis, justification of importance of mechanochemical activation to increase the materials reactivity.

O. V. Matyukhin – processing and visualization of experimental data, plotting the dependence of phase composition on pressure and temperature, analysis of the research results, writing the conclusions, discussion of the results.

М. В. Клеоновский – разработка концепции исследования и постановка задач, проведение экспериментальной части работы, помол и прессование проб при различных давлениях, анализ химического и фазового состава пылей ДСП, а также их изменения в процессе механохимической активации.

О. Ю. Шешуков – научное руководство, разработка методов брикетирования образцов, их обжиг с последующим анализом фазового состава.

М. А. Михеенков – анализ воздействия механохимической активации на фазовый состав пыли ДСП, изучение влияния помола и давления прессования на процессы восстановления оксидов железа, интерпретация данных, полученных в результате рентгенофазового анализа проб и при исследовании взаимодействия компонентов сырьевой смеси.

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