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Prospects for microwave sintering of the composition from waste of the fuel and energy complex

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Abstract. The article considers an option for solving environmental problems associated with high-temperature waste from enterprises of the fuel and energy complex and the metallurgical industry. A technology is proposed for obtaining material from the ash of a thermal power plant, slag waste from metallurgy and a binder from liquid glass by sintering in a microwave field.. Differences in the phase composition, external characteristics and properties of materials obtained by traditional convective firing and sintering in a microwave field are noted. The phase compositions of the fired materials are determined, and the effect of the amount of the amorphous phase on the final properties of the material is noted. The article shows the structures of fractures of materials, shows the difference in the fineness of the structure of the sample based on ash, obtained by convective firing and sintering in the microwave field. Shown amorphous phase between crystalline formations in the structure of the material, marked by X-ray phase analysis. The prospect of microwave sintering of refractory multicomponent compositions in the microwave field to obtain materials is shown.

1. Introduction

Currently, the growing needs of mankind for energy have led to significant environmental problems associated with both emissions of pollutants into the atmosphere, wastewater discharges, and high-temperature waste from enterprises of the fuel and energy complex, the metallurgical industry. These are ash dumps of thermal power plants accumulated over a long period of operation, slag waste from metallurgical production, which create a significant negative impact on the environment in the industrial regions of the country. At the same time, they are a valuable raw material for such industries as ceramic, refractory, cement clinker production. All these are traditionally roasting technologies that use natural fuel and are forced to create an environmental problem due to harmful emissions into the atmosphere.

The growing needs of society for energy resources are forcing the most efficient use of natural fuel and the potential of the energy sector. At the same time, the development of alternative innovative technologies to replace traditional firing technologies for the production of materials is becoming especially significant. This is especially important in technologies for obtaining materials from substances that have already undergone high-temperature processing and consist of high-temperature crystalline phases and glass. These include ash wastes from the heat power industry and slags from metallurgical industries.

Studies of alternative technologies to traditional firing have shown the prospect of using a high-frequency electromagnetic field for sintering raw materials in ceramics [1-3]. It has been shown that sintering in a microwave electromagnetic field makes it possible to obtain microcrystalline materials with increased density and strength. The study of the mechanism of sintering of polymineral compositions in the microwave field showed that the sintering process begins in the region between the grains of the composition. These studies indicated the prospects for the use of substances consisting of high-temperature phases and do not require additional heat treatment [4-11].

Ash and slag waste includes high-temperature crystalline phases, such as mullite, quartz, sillimanite, belite, magnetite, hematite, glass, clay minerals [12-13]. The composition of ashes and slags includes crystalline and amorphous phases. The activity of ash and slag increases with increasing temperature, mechanical grinding and exposure to alkaline components. The strength of the ash-alkali stone is influenced by the mineral composition and the ratio of the crystalline and amorphous phases [12].

In addition, there is a tendency in the power industry to replace environmentally problematic technologies that use natural fuel combustion with an efficient technology for transmitting energy by a microwave beam through the creation of solar space power plants (SSP) [14-16]. The ecological feasibility of using microwave energy in industry for the production of materials and wireless transmission of energy in the energy sector have great prospects for development.

Prospects for the use of microwave sintering in the technology of obtaining firing materials are associated with a high rate of transfer of high-frequency energy when interacting with a substance. At the same time, there is an increase in strength and toughness indicators during the destruction of the material, associated with a decrease in the size of the grain and defect structure of the material [4-11]. A high heating rate leads to an increase in the temperature in the interphase region, acceleration of particle diffusion, acceleration of reactions, phase transformations, and the formation of a microcrystalline structure. For traditionally brittle ceramics, properties of superelasticity and plasticity are manifested [1-3].

The transition to new ecological principles of organizing the production of firing materials is possible with the use of the latest innovative technological developments, the most promising of which can be called heating due to a high-frequency electromagnetic field, which has already found application in various industries. From this point of view, it is of interest to use high-temperature wastes, which include high-temperature phases that were formed earlier in the course of heat treatment. For bonding high-temperature phases, you can try using a heat-resistant binder. In this case, you can get a material with the required performance characteristics.

Previous studies have shown that the multicomponent composition of wastes for sintering in the microwave field presents a certain difficulty due to the different response of each component to the electromagnetic field. The study of ceramic dielectrics - microwave energy absorbers, which include metal oxides, such as: SiO_2, Al_2O_3, CaO, MgO, showed a different reaction of oxides to the action of the microwave field. The ability of oxides to convert microwave energy into heat and dissipate excess energy was noted [17].

Investigation of the sintering process of polymineral aluminosilicate compositions showed that the introduction of a mineralizer enhances the interphase interaction and promotes the formation of an amorphous phase [18]. Previously, it was found that activation in a microwave field of an aluminosilicate composition and subsequent convective firing leads to the formation of a composition of nanoscale formations and increased strength of the material [19-20].

The aim of the work was to investigate the possibility of obtaining a material by microwave sintering of a composition of ash, slag with a binder based on water glass.

2. Materials and methods

This work investigates the process of sintering in a microwave field of samples from compositions based on ash from a thermal power plant, metallurgical slag and liquid glass. A comparison is made of the final structure of the fired material in the microwave field and the structure after convective firing in a muffle furnace.

To study the sintering process, the following components were selected: fly ash from the Novo-Irkutsk CHPP and ferroslag of the Izhevsk Mechanical Plant, containing 40-50% crystalline and 50-

60% amorphous phases. Ash and slag were selected with a similar chemical composition. The chemical composition of ash and slag is presented in table 1 in masses %:

Table 1. Chemical composition of components of mix.

Components	Color	Chemical composition of the main oxides, masses %						
		SiO_2	Al_2O_3	TiO_2	Fe_2O_3 + FeO	CaO +MgO	Na_2O $+K_2O$	Loss on ignition
Ashes	Gray	56.19	24.50	0.61	7.83	5.92	1.43	3.52
Slag	Black	53.13	24.34	1.22	11.29	2. 94	2.59	4.49

The color of ash and slag is determined primarily by iron oxides. In the crystalline fraction of fly ash, iron oxides are represented by black particles of magnetite (Fe_3O_4) mixed with hematite (Fe_2O_3) [21]. The black color of the slag is determined, first of all, by ferrous oxide FeO - wustite [13].

Molding mixtures were prepared from raw materials, ground and sieved through a sieve with a hole diameter of 1 mm. Samples were prepared by plastic molding from a mass obtained after moistening ash and slag with liquid glass with a density of 1.4 g / cm^3. The mass was moistened until a plastic dough (10-12%) was obtained, and specimens with a size of 20 × 20 × 20 mm were molded. Samples without preliminary drying were placed for firing in a muffle and microwave ovens. They were fired in a microwave oven at 1000 °C with holding at a maximum temperature for 5 min. In a muffle furnace up, to a temperature of 1000 °C and holding for 1 hour. The total period of rise and holding at maximum temperature in a muffle furnace is 5 hours, in a microwave oven for 20 minutes.

Microwave treatment was carried out in a microwave oven (Samsung m 1711 NR) at an output radiation power of 800 W at an operating frequency of 2.45 GHz. The magnetic field is created by a 50 Hz power frequency current that flows in the furnace power supply system. For sintering, a muffle made of mullite-silica slabs and kaolin wool is installed inside the furnace. The temperature was controlled by a thermocouple, the junction of which was covered with a radiation-protected coating and installed near the sample.

X-ray phase analysis of the fired samples was carried out on a Shimadzu XRD 6000 diffractometer in CuK-radiation (PDF 4+ base, POWDERCELL 2.4 full-profile analysis program), sampling of sample breaks - on a system with an electron and focused ion beam (Quanta 200 3D) in the Tomsk Regional Center for Shared Use of NU TSU.

3. Results

All samples fired in muffle and microwave ovens were sintered without destruction. Samples fired in a muffle furnace had a terracotta color of fired ceramics. Samples based on ash and slag, fired in a microwave oven, had a gray color, close to the color of ash and slag.

The test results of the samples are shown in the graph in figure 1.

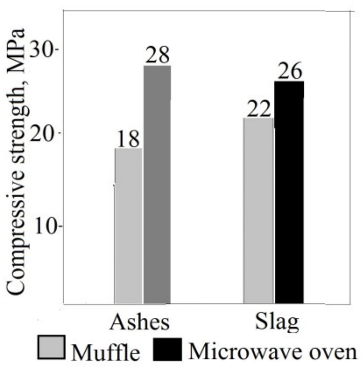

Figure 1. Compressive strength of samples based on ash and slag with water glass.

It can be seen from the graph that the samples fired in the microwave oven had a strength exceeding the strength of the samples fired in the muffle furnace. The strength of the samples obtained on the basis of ash and slag is within the same limits as for the samples fired in the field Microwave, and for samples fired in a muffle furnace. The greatest differences in strength were found for ash-based samples. The phase composition of the samples is shown in table 2.

Table 2. Phase composition of the samples after firing.

Composition	Weight percent	Maintenance of phases, mass%	Crystallite size, nm
Ashes muffle	SiO_2	57.18	-
	$Al_2Si_2O_5$	16.13	-
	Fe_2O_3_HEMATIT	0.76	-
	$(Na_{0,7}K_{0,3})(Al_{1,02}Si_{2,98}O_8)$	25.94	-
Microwave oven ashes	SiO_2	6.9	48.3
	$Al_2Si_2O_5$	9.6	12.8
	Fe_2O_3	0.4	-
	AlSiO_mullite	5.9	43.1
	Amorphous phase, phase traces $CaCO_3$	76	
Microwave oven slag	SiO_2	3,5	-
	AlSiO_mullite	9.6	36.4
	$Al_2Si_2O_5(OH)_4$_kaolin	13.6	167.8
	$NaAlSi_3O_8$	12.8	28.9
	$CaCO_3$	11.5	> 150
	Аморфная фаза, следы фазы Fe_2O_3	49	

According to the data of X-ray phase analysis, the samples fired in a muffle furnace contain crystals of hematite Fe_2O_3, which give the samples a characteristic terracotta color. All phases lie outside the nanoscale level. The sample fired in the microwave field has a mullite phase and a large percentage of the amorphous phase. Crystalline phases are in the nanoscale range. This can explain the high strength of the samples fired in the microwave field. The amorphous glass phase imparts special strength to the product by sticking together refractory ash particles. The composition of the amorphous phase affects the strength of the composition. X-ray diffraction patterns of samples obtained on the basis of ash and slag and fired in the microwave field are shown in figure 2.

A large halo on the X-ray diffraction pattern of a composition based on ash and water glass during sintering in a microwave field is confirmed by decoding in table 2. The composition based on slag and water glass has a lower percentage of the amorphous phase, and the samples showed lower strength during testing (figure 1).

The microstructure of an ash-based alloy obtained by firing in a muffle and microwave oven is shown in figure 3. The coarse-grained structure of the alloy obtained by traditional firing and the dispersed structure of the sample obtained by sintering in the microwave field are visible. The enlarged image shows the amorphous phase present in the composition according to X-ray phase analysis data.

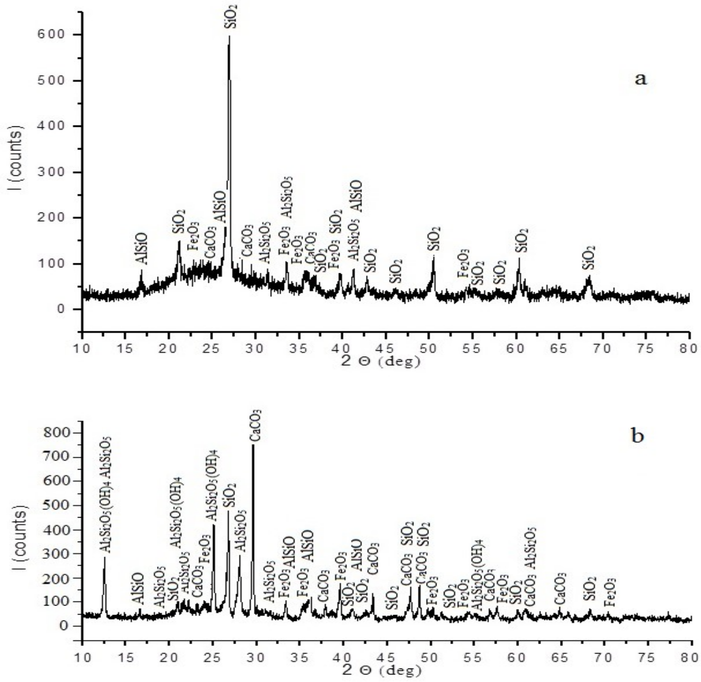

Figure 2. Results of X-ray phase analysis of samples from the composition: ash-water glass (a) and slag-water glass (b) with phase decoding.

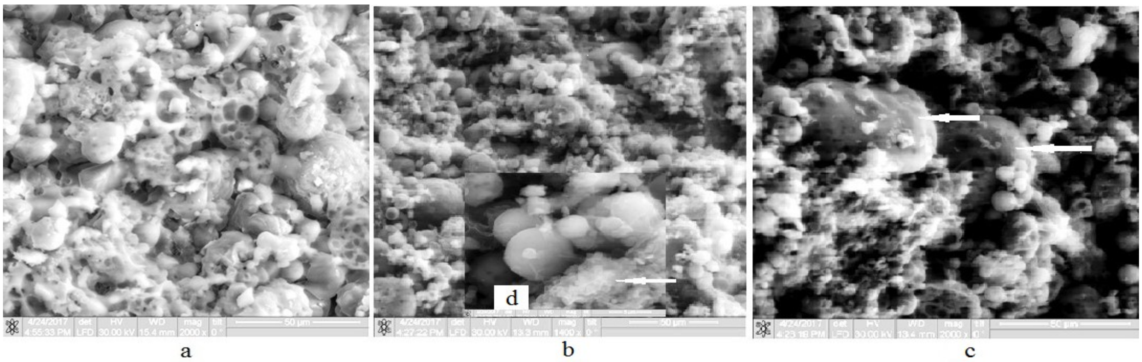

Figure 3. Microstructure of fractures of samples of compositions of compositions: ash with water glass (a, b, d), slag with water glass (c), fired in a muffle (a) and microwave field (b-d).

Figure 3 and table 2 show that the dispersion of the main phases in samples sintered in a microwave field is higher than the microstructure of a sample fired in a muffle furnace. An amorphous phase is visible present in the composition (d). Large inclusions of quartz crystals are seen in the slag-based sample.

Specimens with a dispersed dense structure of the composition and a large amount of amorphous glass phase obtained by sintering in a microwave field show a high strength of the material. This is confirmed by the results of testing samples based on ash, slag and water glass.

4. Discussion

The paper presents an analysis of the literature data on a possible solution to the environmental problem associated with the utilization of waste from the production of heat and power engineering, and promising directions for the development of the energy complex. Utilization of waste from heat power engineering and metallurgy is relevant for many regions of the country, despite the fact that they are a valuable raw material for many areas of industry. Listed are studies on the synthesis of materials in an electromagnetic field, which are distinguished by special, often extreme properties. The results of the study of the technology of synthesis of materials in an electromagnetic field are presented. Materials obtained by sintering in an electromagnetic field are distinguished by extreme properties associated with the peculiarity of the microstructure and the presence of nanoscale phases. Such compositions are characterized by a viscous nature of destruction. This is especially important for the hardening of traditionally brittle ceramics.

A comparative analysis of the structure and properties of materials obtained by traditional convective firing and sintering under conditions of a high-frequency electromagnetic field has shown the possibility of obtaining a material with nanoscale phase dispersion and high strength. The mechanism of high-speed sintering in a microwave field implies the possibility of utilizing refractory production wastes to obtain materials for various purposes. Since silica is the basis of most polymineral materials, it is assumed that it is rational to use water glass to bind refractory components in a microwave field, when the sintering process begins in the interfacial region.

The study of firing technology in electromagnetic fields creates prospects for solving the environmental problem of firing technologies in metallurgy and the ceramic industry, the development of materials science in the direction of innovative environmental technologies for obtaining materials with special properties. The research carried out in this direction is important for the development of the fundamental principles of the technology for obtaining materials in general, and especially from polymineral raw materials.

The presented work is devoted to the study of the possibility of using refractory technogenic waste from heat power engineering and metallurgy to obtain materials with high strength characteristics under conditions of firing in a microwave field. Liquid glass was considered as a binder.

5. Conclusion

The result of the study was the process of sintering samples from ash and metallurgical slag with liquid glass in a microwave electromagnetic field.

As a result of the research it was established:

- The fundamental possibility of sintering in the microwave field of a polymineral composition of refractory compounds that are part of ash and slag, with a binder made of liquid glass;
- The dependence of the sintering process and material properties on the composition of the raw powder composition;
- The difference in the phase composition and dispersion of the composition sintered in the microwave field from the composition of the identical composition, traditionally fired;
- Formation of a nanoscale phase during sintering in the microwave electromagnetic field, the composition of which depends on the composition of the raw material composition;
- The amount of amorphous phases during sintering in a microwave field depends on the composition of the raw material composition and affects the final properties of the material.

The results of the study on the sintering in the microwave field of polymineral compositions based on ash and slag with liquid glass showed the prospect for the development of a technology for the utilization of technogenic refractory waste from heat power engineering and metallurgy and the possibility of obtaining firing materials.

The results obtained coincide with the conclusions made earlier on sintering of refractory compounds and polymineral mixtures. An increase in the strength of the material is associated with the phase composition and the formation of a special structure of the material, the presence of nanoscale phases.

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