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Green technologies in energy and materials science - development prospects

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Abstract. Literature information about the problems in the technology of energy and ceramics is given. In the article the information about the promising directions of development of energy and firing technologies, about the influence of sintering material in the microwave field on the structure and properties of the material and the prospects of this direction in the hydrogen energy industry is considered. The results of firing samples of diatomite with additives of carbonate clay (marl) and NaCl salt in a microwave oven are considered. Microphotographs of the structures of the obtained materials are presented. For compositions of diatomite, a mixture of diatomite, marl and NaCl salt, the results of X-ray diffraction analysis and microphotographs of material structures are considered. For diatomite containing montmorillonite clay, a large exothermic effect in the temperature range 400 - 700 ° C was found upon heating. The composition based on diatomite with the addition of marl and salt passes this temperature range with endothermic effect. A promising direction of research into the technology of synthesis and storage of hydrogen is shown.

1. Introduction

The exploration of space and nuclear power, the overarching environmental problems of our time have fostered increased research into new environmental technologies for firing materials and the generation of renewable energy as an alternative to carbon-based fuel technologies.

The needs of space, nuclear power in materials with special properties, often unattainable by traditional technologies, as well as advances in nanotechnology, have led to studies of the possibility of using for the needs of energy and materials science, especially as the energy of thermal processes the energy of the electromagnetic field.

The basis for a real contribution to the energy of this idea was the results of research by American scientist P.E. Glaser to collect solar energy and transfer it by a beam of microwave radiation to the surface of the. Earth and wirelessly deliver it to the consumer. The reality of this technology was confirmed by the results of research at the Space Research Institute in Japan, the project SCES 2000 (SPS 2000) [1].Russian scientists have proposed the use of ceramic dielectrics for wireless energy transfer [2].

At the same time, nanotechnology researchers obtained results on sintering compositions in an electromagnetic field. The materials obtained by this technology showed properties that could not be achieved by conventional convective firing: these were, first of all, increased strength, hardness, and toughness, for which the nanoscale structure of the material was responsible [3-7].

The studies obtained on sintering of simple substances in the microwave field were continued for complex compositions and, especially important, was the study of natural polymineral compositions - hydroaluminosilicates, widespread on earth and in space.

Thus, a great innovative development potential was made on the study of the use of microwave - energy in the energy industry, including wireless power transmission, which has significant advantages over traditional technologies of transporting electricity to the consumer [7].

An encouraging prospect for hydrogen energy was noted after the discovery of the possibility of obtaining and accumulating hydrogen in aluminosilicates after their treatment in the microwave field [8-9].

The speed of interaction with matter is the most important parameter of the electromagnetic field. Acceleration of charged particles movement increases the temperature difference in the interphase region. This leads to an increase in the reaction rate, reduction of grain size and structure defects, leads to the formation of the phase structure of the material of increased hardness and toughness at fracture [3-7].

When ceramic materials are fired in an electromagnetic field, such properties as superelasticity and plasticity, which are not characteristic of them, appear [10].

The results of using the ceramic material as a solid ion-conducting electrolyte, which can work up to 1000 °C, showed the prospects of using this method to produce a material whose synthesis is accompanied by the formation and accumulation in its structure of hydrogen [11].

Fulfillment of this condition is possible if during absorption of any type of energy hydrogen can be released due to the splitting of water present in the structure of the initial composition. In this case, the material must not lose its strength. These conditions are satisfied by natural hydroaluminosilicates, the mineral component of which contains crystallization water along with adsorption water, which escapes during the splitting of the mineral at temperatures up to 600 °C, depending on the type of mineral.

High-temperature electrolysis of water vapor accompanied by hydrogen release at the cathode can take place in cells made of solid oxide-based electrolyte, which has conductivity due to the transfer of oxygen ions formed during water dissociation [11-12].

In this direction, there are developments of ceramic materials, which are used as absorbers of electromagnetic field energy, which include basic oxides of clay minerals [13]. Taking into account the oxide composition of materials, clay-carbonate mixtures, such as marls, may be of the greatest interest from natural compositions.

Earlier studies of clay activation in an electromagnetic field showed the formation of a dispersed structure already at the stage of activation and in the sintered material nanoscale structure [13-14]. The presence of carbonates in the clay composition provides a porous structure of the shingles [15].

Considering the mechanism of sintering in the electromagnetic field [16] and the results of studies on sintering of basic clay compositions [15], the presence of flux is necessary to obtain high-quality material as part of the mass. In this case, a fusible mass is formed between the grains, which, when cooled, forms a strong framework of porous material.

The aim of the work was to analyze the energy activity of clay-carbonate compositions during heating in a high-frequency electromagnetic field.

2. Materials and methods

Based on previous studies, as well as to implement the purpose of studying the energy activity of the system in the process of obtaining a microporous structure of the material, the following raw materials were selected. As the clay-carbonate composition - marl (clay Maximkovskogo deposits) composition (wt. %): 33.2% SiO2, 11.4% Al2O3, 26.1% CaCO3 + MgCO3, 3.6% Fe2O3, 3.6% Na2O + K2O. As an amorphous silica - diatomite deposits Inza composition (wt. %): 90.2% SiO2, 5.35% Al2O3, 0.8% CaO + MgO, 3.22% Fe2O3, 0.13% Na2O + K2O. Salt NaCl was used as an activator for sintering. The mineral composition of the marl is represented mainly by montmorillonite clay and highly dispersed calcium and magnesium carbonates [17]. Diatomite consists mainly of amorphous silica (70%), montmorillonite clay (14%), mica (7%), and quartz (8%) [18].

The process of preparation of the molding sand includes mechanical crushing of the initial components.

Clay-silica compositions are finely dispersed mixtures of hydrated aluminosilicates of different mineral composition, moisture and technological parameters. Due to these differences the compositions after firing show a great variety of final properties of products. Application of fluxing additives - salts of alkali metals, allows to regulate final properties of a material due to activation of ion-exchange processes on border of an interface of phases of mineral particles [13].

Preparation of the molding mixture began with grinding all the components of the future composition to a grain size of less than 1 mm. The powder was moistened with water or salt solution to obtain a plastic dough and molded samples, which were fired in a microwave oven. The samples were kept in the microwave oven at the maximum temperature rise rate of 30 min. The samples were incubated at a maximum temperature of 1000 °C for 5 min.

We used a microwave oven (Samsungm 1711 NR) for sintering. A muffle made of mullite-silica plates was installed inside the furnace. The temperature was controlled by a thermocouple, the junction of which was installed near the sample and protected from radiation by a coating.

X-ray phase analysis of the annealed samples was performed on a Shimadzu XRD 6000 diffractometer in CuK - radiation (PDF 4+ database, full-profile analysis program POWDERCELL 2.4), fracture imaging of samples - on a system with electron and focused ion beam (Quanta 200 3D) in Tomsk Regional Center for Shared Use of NU TSU.

3. Results

In the process of studying the effect of microwave fields on clay-silica compositions, an increased response of silica to the electromagnetic field by deformation oscillations of interatomic bonds was noted [13].

To study the energy activity of the composition during sintering under microwave field conditions the composition based on marl, diatomite and NaCl salt was selected. The activity of silica and previously obtained data on the qualitative sintering in the electromagnetic field of samples based on silica-carbonate compositions were taken into account.

The results of differential calorimetric analysis (DSC) of the compositions are shown in figure 1.

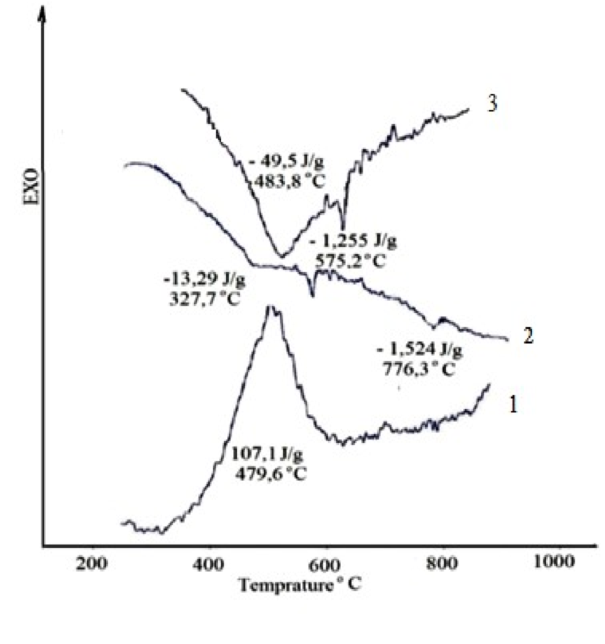


Figure 1. Differential and thermal curves of compositions on the basis of diatomite – 1, with NaCl salt inclusion – 2, with inclusions of NaCl and marl – 3.

Fragments of thermograms are given in the temperature range of decomposition of mineral components of the studied compositions with overlapping processes of removal of strongly bonded water from the clay-silica part of the system, recrystallization of silica, decomposition of montmorillonite and carbonate component of the composition [17-18]. In the same temperature range is possible rearrangement of the crystal lattice and amorphization of clay minerals, the phase transition of amorphous silica in the low-temperature crystalline form of β-quartz and loss of crystallization water by montmorillonite [18]. For the diatomite-based composition, a large exothermic effect can be seen in the temperature range in which the formation of high-temperature phases begins. When increasing the amount of marl in the diatomite there is an endothermic process, going with the absorption of energy, which is wasted on the decomposition of the initial components. The presence of flux leads to the formation of the liquid phase and the passage of heating without significant exo- and endo- processes in figure 1.

The exothermic process of phase formation during diatomite heating is accompanied by the release of energy. This process may be associated with the passage of heterogeneous-catalytic reactions at the interface in the porous structure of diatomite [8; 11; 16].

Between the layers in the montmorillonite structure it is possible to conduct reactions to obtain hydrogen during the interaction of the active metals Al, Mg with water [19]. The probability of this process is justified by the results of research on the extraction of metals from polymineral compositions by microwave radiation [20]. Since the technology of hydrogen accumulation is based on the need to form a porous structure in the sintering process [21]. This process is facilitated by the reaction of water to microwave irradiation [22] and the processes possible in the space between the layers of clay minerals during microwave irradiation, identical in SOFC.

X-ray phase analysis of the diatomite-based sample showed the presence of crystalline quartz and iron oxide, the formation of which occurs in the temperature range of decomposition of clay minerals, the yield of crystallization water and the possible formation of hydrogen. The strength of the sample is comparable to that of the sample based on marl, diatomite and NaCl salt due to the formation of a composition of crystalline phases. The results of the strength test of the specimens are shown in figure 2. It can be seen, that the strength of all specimens is within the same limits.

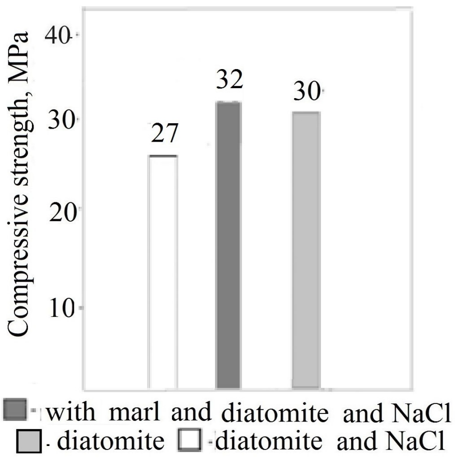
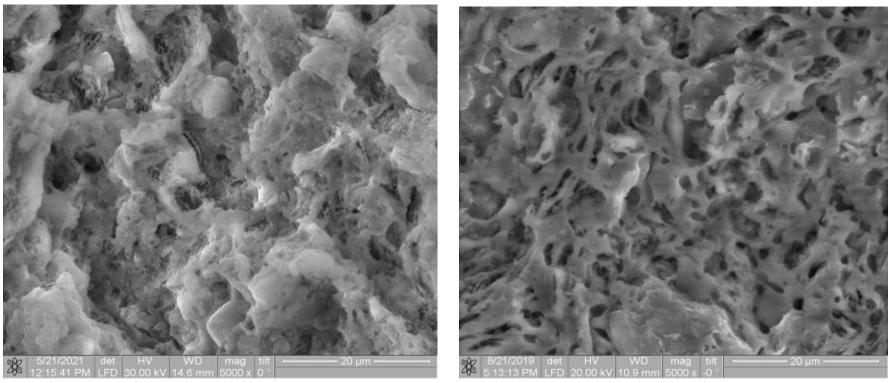


Figure 2. Strength of samples based on diatomite with marl and NaCl salt additives after firing at 1000 °C.

For compositions based on diatomite the porous structure of the sintered material is realized. For the composition based on marl a dense structure with glass-phase content of about 40% is realized, shown in figure 3 and table 1.



a b

Figure 3. Fracture micrographs of samples of burnt diatomite (a) and a mixture of diatomite with marl and NaCl salt (b).

Table 1. Phase composition of the samples after firing.

Composition	Weight percent	Maintenance of phases, mass%	Crystallite size, nm
Marl, diatomite, NaCl	$Ca_2MgSi_2O_7$	21.6	12.4
	SiO_2	17.6	51.8
	$CaMgSi_2O_6$	24.6	15.2
	Ca_2Mg0,08Al,84Si1,08O_7 Traces of the amorphous phase	< 40	
Diatomite	SiO_2	50.0	> 200
	$Al_2Si_2O_5$	18.7	29.8
	γ-Fe_2O_3	3.4	> 200
	$Na_{0.7}K_{0.3}Al_{1.02}Si_{2.98}O_8$	28.0	28.7

4. Discussion

The paper analyzes the possibility of using the microwave electromagnetic field in promising areas of energy and environmental technologies for obtaining sintering materials. The analysis of energy activity of clay-carbonate compositions, which showed high strength and quality of the material during sintering under the conditions of heating in the microwave field, is given.

Analysis of energy processes in the temperature range of 400-700 °C, which undergo clay-silica mineral components, of the mixture corresponds to the superposition of processes of removal of strongly bound water from the clay-silica part of the system, recrystallization of silica, decomposition of montmorillonite and the carbonate component of the composition [17-18], as well as the possible passage of reactions to obtain hydrogen in the interaction of the active metals Al, Mg with water, the pairs of which may form in the structure of montmorillonite during its decomposition and amorphization [19].

These processes can be confirmed by a large exothermic effect and energy release in diatomite, the clay part of which includes montmorillonite.

Crystalline quartz, present in the composition, reacts to the microwave field by deformation oscillations of the bond, contributing to the reactivity of the entire system [13]. This can indirectly confirm the assumption about the possible release of hydrogen.

5. Conclusion

The paper presents the results of studies of the firing process in a high-frequency electromagnetic field of natural amorphous silica (diatomite) and compositions with clay inclusions of Ca, Mg carbonates - marl and fluxing salt NaCl.

It was found that the heating of diatomite in the temperature range 400 - 600 °C is accompanied by a large energy release (107.1 J/g) and exothermic process. This process may be associated with crystallization of quartz [18] and possible decomposition of water molecules with the release of hydrogen.

Microphotographs of the structure of burnt diatomite show micron-sized pores.

The composition of diatomite with fluxing salt NaCl when heated is not detected exothermic process.

Sintering of diatomite with large amounts of marl and fluxing salt in the temperature range 400-600°C, in which hydrogen formation is possible, occurs with a large endothermic process associated primarily with the decomposition of the carbonate component of marl and the decomposition of clay minerals.

After sintering, the composition of diatomite with the addition of marl and salt has a dense structure in the fracture. X-ray phase analysis shows the presence of an amorphous (glass) phase.

The strength of diatomite samples and compositions with additives is within the same limits.

The peculiarities of temperature processes during heating and the final structure of the material depend on the amount of carbonate clay in the composition of diatomite.

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