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Mechanochemically Stimulated Synthesis and Low Temperature Sintering of MgAl_2O_4

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Abstract

A sequence of changes in X-ray patterns on heating of MgAl_2O_4 precursors synthesized by different methods is considered. It is shown that the presence of only spinel phase peaks can cause the erroneous conclusion of a single-phase nature of the product and thus explain its low sinterability. Mechanochemical treatment of the stoichiometric mixture of aluminum and magnesium hydroxides yields precursors, which form the powders of single-phase spinel with a crystallite size of 15–30 nm (depending on the composition of the gas medium) when heated at 1000 °C for an hour. Additional disaggregation of spinel powders through low-intensity mechanical treatment (10g, 5 min) in ethanol gives rise to increased sinterability and allows attaining 95 % density at no more than 1350 °C, which is sufficient for successive densification by means of hot isostatic pressing. However, vacuum sintering is less efficient, leading to low density due to residual alkoxy groups enclosed in gas-tight aggregates which are formed in the course of the treatment with ethanol.

Keywords: magnesium aluminate spinel, mechanosynthesis, sintering

INTRODUCTION

Polycrystalline ceramics composed of aluminum-magnesium spinel is widely used in metallurgy, mechanical engineering, automobile production, electrical and radio engineering, and since recent years also in the defense industry. This is provided by a combination of such properties as a high melting point, mechanical strength, low dielectric loss, stability to chemically aggressive media, and transparency within wavelength range 0.2–6 μm [1–3]. At the same time, many of these properties are critically dependent on residual porosity in sintered material and on the parameters of its microstructure: the lower is porosity and the smaller is the size of grains in the ceramics, the higher are strength and transparency of the material [4]. It is impossible to obtain ceramics with minimal porosity and at the same time with the conservation of grain size within

the submicron range without using ultrafine spinel powders allowing the formation of dense material at relatively low temperature which does not cause substantial growth of grains. These considerations stimulated the development of alternative methods of the synthesis of spinel nanopowder [5] because a 100 % yield of spinel from traditional solid-phase synthesis was achieved only at a temperature within the range of 1500–1600 °C [1], while particle size was equal to several ten micrometers. Subsequent long-term intense grinding of spinel caused its contamination with the material of milling bodies and still provided the density equal to 99.5 % only at 1650 °C, with grain size at a level of 10 μm [6].

The mechanochemical method of the synthesis of nanodispersed spinel allows one to decrease the temperature of its synthesis to 750–850 °C, with crystallite size down to 6.6 nm [11], and even to conduct the synthesis at room temperature

[13]. However, in spite of nanometer size of crystallites, as determined from line broadening in the diffraction patterns, one succeeds in achieving 97–98 % density only at a temperature of 1550–1600 °C [9, 10], and the average grain size at this temperature exceeds 50 μm [14]. A possible reason for unsatisfactory sintering of the described spinel powders is their aggregated state, as well as the presence of the traces of individual oxide phases [8]. Indeed, it was indicated in [11] that the specific surface of the powder with crystallite size 6.6 nm is only slightly larger than 2 m^2/g , which corresponds to ~800 nm as calculated for the size of spherical particles, that is, the powder is strongly aggregated, and the aggregates are gas-tight. On the other hand, it follows from the difference of the theoretical densities of aluminium oxide, magnesium oxide and spinel (3.98, 3.58 and 3.58 g/cm^3 , respectively) that the formation of spinel from Al_2O_3 and MgO should be accompanied by volume expansion ~8 %. Because of this, if the sample after synthesis contains residues of individual oxides, they will interact with each other at sintering temperature higher than the synthesis temperature, and this will cause local distortion of particle packing and the formation of hard-to-heal defects.

To conserve the size of spinel grains in the submicron range, it is necessary to sinter the ceramics at a temperature not higher than 1450 °C [4, 14], and the achievement of high density is provided by means of hot isostatic pressing (HIP). This method may be applied only after preliminary sintering of the ceramics, leading to pore closing in the material, which usually happens at a density >90 % of the theoretically possible value, so it is necessary to obtain the powders providing this level of density after preliminary sintering within temperature range 1300–1350 °C, after which further densification of the material by means of HIP is possible until a transparent material is obtained.

In the present work, we made an attempt to eliminate the above-indicated reasons of low activity to sintering and to implement the potential of spinel nanopowders synthesized mechanochemically, for obtaining the ceramics with high density at relatively low temperature.

EXPERIMENTAL

The reagents used in the work included aluminium hydroxide $\text{Al}(\text{OH})_3$ (GOST 118418–76), magnesium hydroxide $\text{Mg}(\text{OH})_2$ (TU 6-09-

3759–86), aluminium nitrate nonahydrate $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (GOST 3757–75), magnesium nitrate hexahydrate $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (GOST 11088–75), ammonium hydroxide NH_4OH (Os. Ch. grade, GOST 24147–80), $\alpha\text{-Al}_2\text{O}_3$ nanopowders with the average particle size equal to 20 (α_1) and 50 nm (α_2), obtained according to the procedures described previously [15, 16].

Co-precipitation was carried out by adding NH_4OH drop by drop into the solutions of aluminium and magnesium nitrates with a concentration of 2.8–3.0 mol/L till the formation of a gel-like precipitate at pH 8. The precipitate was washed and then annealed in the air at a temperature of 300 °C to the point when nitrate decompositions topped.

Spray drying was carried out by spraying the solution of magnesium and aluminium nitrates with the concentration 2.3 mol/L at the chamber temperature 160 °C with the help of Mini Spray Dreer B-290 installation (BÜCHI Labortechnik AG, Switzerland).

Mechanical activation (MA) of the mixtures containing $\text{Al}(\text{OH})_3$ and $\text{Mg}(\text{OH})_2$, or $\alpha\text{-Al}_2\text{O}_3$ and $\text{Mg}(\text{OH})_2$ in the amounts calculated for obtaining spinel MgAl_2O_4 with the stoichiometric composition was carried out in an AGO-2M mill (Russia) equipped with a rotational speed governor. The ratio of loaded mass to ball mass was 25 : 1, the balls 3 mm in size and cylinder walls were made of stabilized zirconium dioxide.

The obtained precursors were annealed at different temperatures (T_{ann}) in a LAC VP 04/17 high-temperature furnace (Czechia) (or Carbolite STF 15/180 (Great Britain), with water vapour blown through the furnace if it was necessary to make an atmosphere with increased humidity). The heating rate was 5 °C/min, isothermal exposure lasted for 1.5 h.

The obtained powders were pressed in tablets 16 mm in diameter, 3–4 mm high, by means of dry uniaxial pressing at a pressure of 30 MPa, followed by isostatic compression at a pressure of 200–250 MPa using an AIP3-12-60C installation (American Isostatic Press, USA). After pressing, the samples were sintered in the air in a LAC VP 04/17 furnace with a heating rate of 5 °C/min and isothermal exposure for 1.5 h. Vacuum sintering was carried out in the SNVE-1,7.3.1,7/20 furnace (LC Prizma, Russia) at a pressure of 10^{-3} – 10^{-4} Pa. Hot isostatic pressing of preliminarily sintered samples was carried out in a graphite furnace for 1 h in argon at a pressure of 200 MPa using the AIP6-30H installation (American

Isostatic Press, USA). The density of the resulting ceramics was determined by means of isostatic weighing according to GOST 20018–74.

For some spinel powders synthesized in the work, disintegration by means of mechanical treatment in ethanol or isopropanol with the acceleration of 10g for 5 min was carried out before pressing.

X-ray phase analysis (XPA) of powders and determination of crystallite size were carried out with the help of a D8 Advance diffractometer (Bruker, Germany) with a step of 0.02° over 2θ and accumulation time 0.2 s. Crystallite size in powders and grain size in sintered materials were calculated using the PowderCell 2.4 or TOPAS software with the compulsory introduction of the parameters of standard samples obtained under identical recording conditions. Results for both programmes coincided with the accuracy of several nanometres. The phase ratio was determined with the help of PowderCell 2.4 software.

RESULTS AND DISCUSSION

It has been already mentioned above that one of the reasons determining the necessity of high

temperature to obtain dense ceramics from nanopowder is the possible presence of a small amount of unreacted individual oxides in the synthesized spinel. All researchers who published their results on the low-temperature synthesis of spinel made conclusions concerning reaction progress only relying on XPA. However, intense MA of the reagents causes their amorphization, and subsequent thermal treatment of the precursor at insufficiently high temperature may lead to the formation of a material with diffraction patterns containing only the reflections of the phase which had been the first to crystallize, thus making the illusion of obtaining a single-phase material. Moreover, due to substantial broadening at low temperatures, the reflections related to the phases that are present in small amounts may be hidden in the reflections of a prevailing compound. To verify this assumption, we synthesized precursors from the solution containing aluminium and magnesium nitrates in the amounts necessary for the formation of the stoichiometric magnesium aluminate spinel through co-precipitation of hydroxides and spraying drying. Then these precursors were annealed at different temperatures (Fig. 1 and 2).

The diffraction pattern of the sample obtained by means of co-precipitation and annealed at

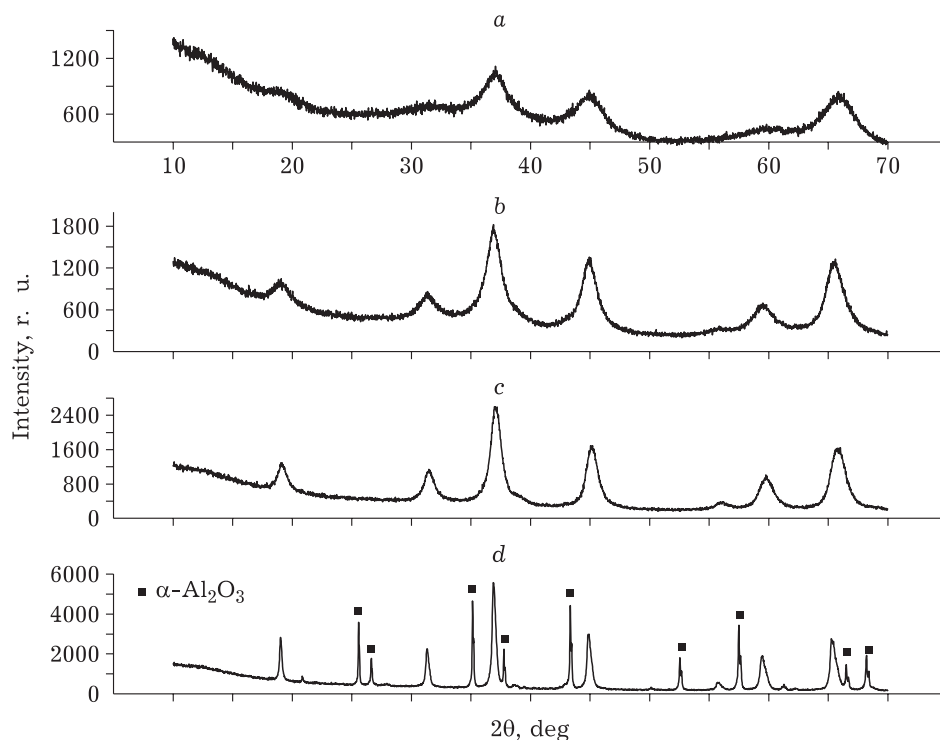


Fig. 1. Diffraction patterns of the product co-precipitated from the solution of magnesium and aluminium nitrates and then annealed at 600 (a), 750 (b), 900 (c) and 1100 °C (d).

600 °C contains only reflections relating to the MgAl_2O_4 phase; these reflections get narrower with temperature rise (see Fig. 1, a–c).

According to calculation results, the average size of crystallites increases with an increase in annealing temperature and becomes equal to 3, 5, 9.5 and 20 nm at $T_{\text{ann}} = 600, 750, 900$ and 1000 °C, respectively. For the sample annealed at 1000 °C, specific surface area equal to $69 \text{ m}^2/\text{g}$ was obtained. Calculated for the size of spherical particles, this value corresponds to ~ 24 nm, which is in good agreement with XPA data. A complete illusion arises that we have got a set of powders of single-phase spinel with different crystallite sizes. However, with an increase in T_{ann} to 1100 °C, a material is formed for which the diffraction patterns point to the presence of $\sim 30\%$ $\alpha\text{-Al}_2\text{O}_3$ in the sample (see Fig. 1, d). A thorough analysis of diffraction patterns showed that starting from $T_{\text{ann}} = 750$ °C, spinel reflections are gradually shifted towards larger angles. It is probable that co-precipitation of a mixture of aluminium and magnesium nitrates with ammonium hydroxide at pH 8 does not provide complete precipitation of magnesium hydroxide, so that after washing the gel-like precipitate, the sample contains aluminium hydroxide in excess. During heating, spinel is the first to crystallize, and it may be concluded from XPA data that a single-phase prod-

uct is obtained. $\gamma\text{-Al}_2\text{O}_3$ is formed after annealing at a higher temperature, but its crystallization may be detected only from a slight shift of spinel reflections to larger angles because the oxide has a similar lattice with slightly smaller parameter and hence close positions of reflections in diffraction patterns. Only at $T_{\text{ann}} = 1100$ °C, when $\alpha\text{-Al}_2\text{O}_3$ with a completely different crystal lattice and the corresponding different diffraction pattern is formed, it becomes evident that there is no single-phase product.

Even in the case when the stoichiometry is not distorted during the synthesis of the precursor, as, for example, in the case of its synthesis by means of spray drying it turned out that the presence of spinel reflections alone in the diffraction patterns of the annealed product cannot serve as the proof of successful synthesis. After annealing of the precursor at 750 °C, its diffraction pattern demonstrates only the presence of MgAl_2O_4 (see Fig. 2, a) with the average crystallite size 5.5 nm, but with temperature rise by only 50 °C we detect the MgO phase, which was completely invisible before (see Fig. 2, b). After $T_{\text{ann}} = 1000$ °C, spinel synthesis is completed (see Fig. 2, c), and only in this case, we may state the presence of a single-phase product suitable for obtaining dense ceramics.

As a consequence, the material formed from the product obtained at $T_{\text{ann}} = 800$ °C (see Fig. 2,

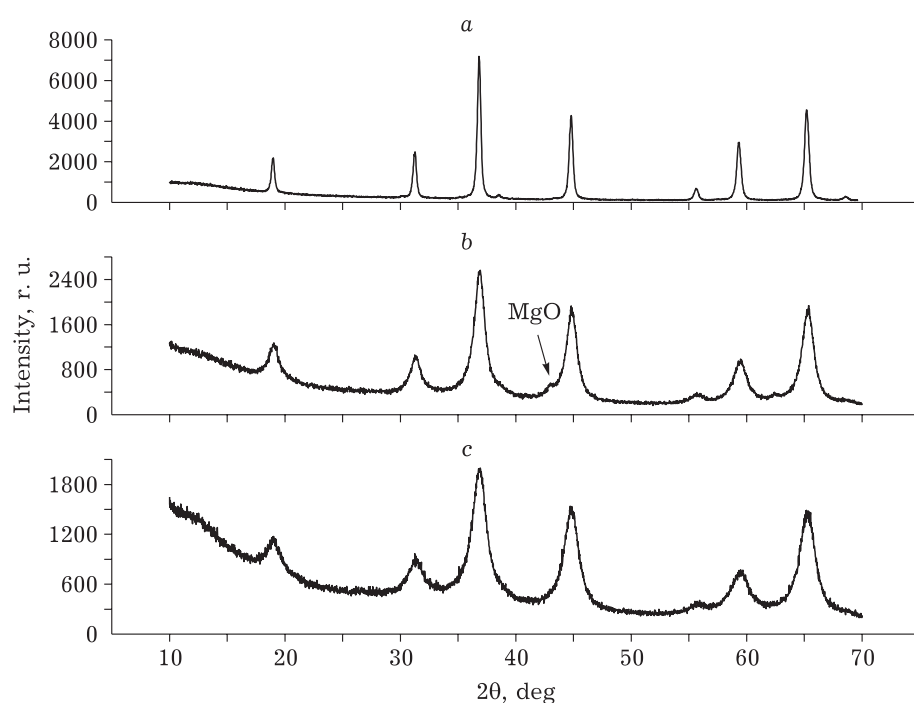


Fig. 2. Diffraction patterns of the product obtained by spray drying from the solution of magnesium and aluminium nitrates and then annealed at 750 (a), 800 (b) and 1000 °C (c).

b) is sintered in the air at 1350 °C only to the density of 73.5 %, while after the synthesis is complete (see Fig. 2, c), in spite of larger crystallites (26 nm), the density of 92.5 % is achieved under the same sintering conditions. It is clear that the use of the product composed of supposedly single-phase spinel with the average crystallite size 5.5 nm all the more would not allow achieving high density during its consolidation.

So, it may be stated that one of the reasons of the low activity of the fine powders of single-phase (according to XPA data) spinel towards sintering is the presence of the residues of unreacted individual oxides, which may be detected only by studying the changes in the diffraction patterns of the sample depending on the temperature of its annealing.

The first stage of mechanochemical synthesis involves grinding of the reagents, then the destruction of lattice to X-ray amorphous state, and only after that components mixing proceeds at the atomic-molecular level. If the intensity of mechanical action is sufficient, the process may finish in product crystallization, which may have been observed in [13]. Intense and long-term MA leads to a strongly aggregated state of the substance [17], which has a negative effect on ceramics sintering [18, 19], therefore, it is reasonable to use the minimal possible time and intensity. In this case, the distribution of components in the precursor would not be homogeneous, so spinel

crystallization under heating will proceed in some regions at a lower temperature, while in other regions, less homogeneously mixed, it will proceed at a higher temperature, while in some regions magnesium and aluminium oxides will be crystallized, and the latter may be not observed in the diffraction patterns as we have demonstrated above. An attempt to obtain dense ceramics from this product will probably fail due to volumetric expansion, which accompanies the synthesis of spinel from oxides. Because of this, before using the synthesized spinel in molding and sintering of the material, it is necessary to check if the obtained powder is truly single-phase and the temperature of precursor annealing is optimal for the 100 % interaction of the components.

The conditions of the mechanochemical synthesis of the precursors of magnesium aluminate spinel and the density of ceramics obtained from the synthesized spinel are presented in Table 1. Before ceramic molding and sintering, the true single-phase nature of the product was confirmed by means of the analysis of the diffraction patterns during gradual heating of the product to 1300 °C.

We did not succeed in obtaining single-phase spinel heating the precursor synthesized at low intensities of MA of a mixture of $Mg(OH)_2$ with nano-dispersed $\alpha-Al_2O_3$ up to $T_{ann} = 1200$ °C. The abrasive properties of aluminium oxide causing the wear of milling bodies did not allow an increase in treatment intensity. A positive result

TABLE 1

Transformation degree of mechanically activated mixtures and the density of sintered ceramics

Reagent	Treatment conditions	Transformation degree during annealing, %		Average crystallite size, nm	Density after sintering, %		
		Temperature, °C			Temperature, °C		
		1000	1200		25	1300	1600
$\alpha_1-Al_2O_3+Mg(OH)_2$	Mortar		51	95			
$\alpha_2-Al_2O_3+Mg(OH)_2$	Mortar		85	90			
$\alpha_1-Al_2O_3+Mg(OH)_2^*$	10g, 10 min		96	80		70	
$Al(OH)_3+Mg(OH)_2$	5g, 10 min		<50	–			
$Al(OH)_3+Mg(OH)_2$	10g, 10 min		98	65			
$Al(OH)_3+Mg(OH)_2$	20g, 10 min		100	70	52	61	93 (HIP)
$Al(OH)_3+Mg(OH)_2$	15g, 10 min	99		25			
$Al(OH)_3+Mg(OH)_2$	20g, 10 min	100		28	55	63	87 (vac)
$Al(OH)_3+Mg(OH)_2^*$	20g, 10 min	100		30	51	83	
$Al(OH)_3+Mg(OH)_2^*$	20g, 10 min	100		30	51	71 (vac)	
$Al(OH)_3+Mg(OH)_2$	20g, 10 min	100 (H ₂ O)		15	47	65	93 (vac)
$Al(OH)_3+Mg(OH)_2^*$	20g, 10 min	100 (H ₂ O)		15	53	93	97 (vac)

Note. $\alpha_1-Al_2O_3$ and $\alpha_2-Al_2O_3$ are nanopowders with average particle size ~20 and ~50 nm, respectively; (H₂O) – annealing in water vapour; (vac) – sintering in vacuum; (HIP) – sintering with the help of HIP.

* Pressing was preceded by disaggregation in ethanol.

was achieved after MA of the mixture of hydroxides with the acceleration of 20g developed in the mill. The heating of the activated mixture leads to the formation of nano-dispersed powder of magnesium aluminate spinel, and the size of crystallites comprising the material may be controlled by varying the synthesis temperature. It should be stressed that in the case if spinel synthesis is carried out in water vapour (see Table 1) to decrease the aggregation of the product, as it was successfully performed for $\alpha\text{-Al}_2\text{O}_3$ in [20], this causes a decrease in crystallite size approximately by a factor of 2.

In spite of the nanometer size of the crystallites comprising spinel, sintering within the temperature range of interest (1300–1350 °C) does not allow us to achieve the required density. Subsequent application of high-temperature vacuum sintering or even HIP is also insufficiently effective. This is evidently connected with the aggregation nature of powders, because oxygen bridges are formed between the neighbouring particles when the surface hydroxo groups are removed at the temperature of spinel synthesis.

If pressing is preceded by the disaggregation of powders obtained at 1000 °C, the density of

ceramics sintered at 1300 °C is substantially higher, and it reaches 93 % for samples annealed in water vapour (see Table 1). Moreover, if we rise crystallization temperature by only 50 °C, spinel powder with the average crystallite size 37 nm after disaggregation allows us to achieve ceramic density 95 % with almost zero open porosity after sintering in the air at 1350 °C. This ceramics is potentially suitable for the application of the HIP method to obtain absolutely pore-free and possibly transparent material.

However, it seems reasonable to use HIP in the case of the material sintered in vacuum, so that gas present in pores would not create pressure impeding pore contraction. One can see in the data presented in Table 1 that sintering in vacuum at 1300 °C leads unexpectedly to a substantially lower density than sintering in the air: 71 and 83 %, respectively. The density of ceramics sintered afterwards in the air at the same temperature (81 %) is comparable with the density of the material sintered only once. It is assumed that non-evaporated surface alkoxy groups that were formed during disaggregation in alcohol are the source of carbon appearing during vacuum thermal treatment and prevent

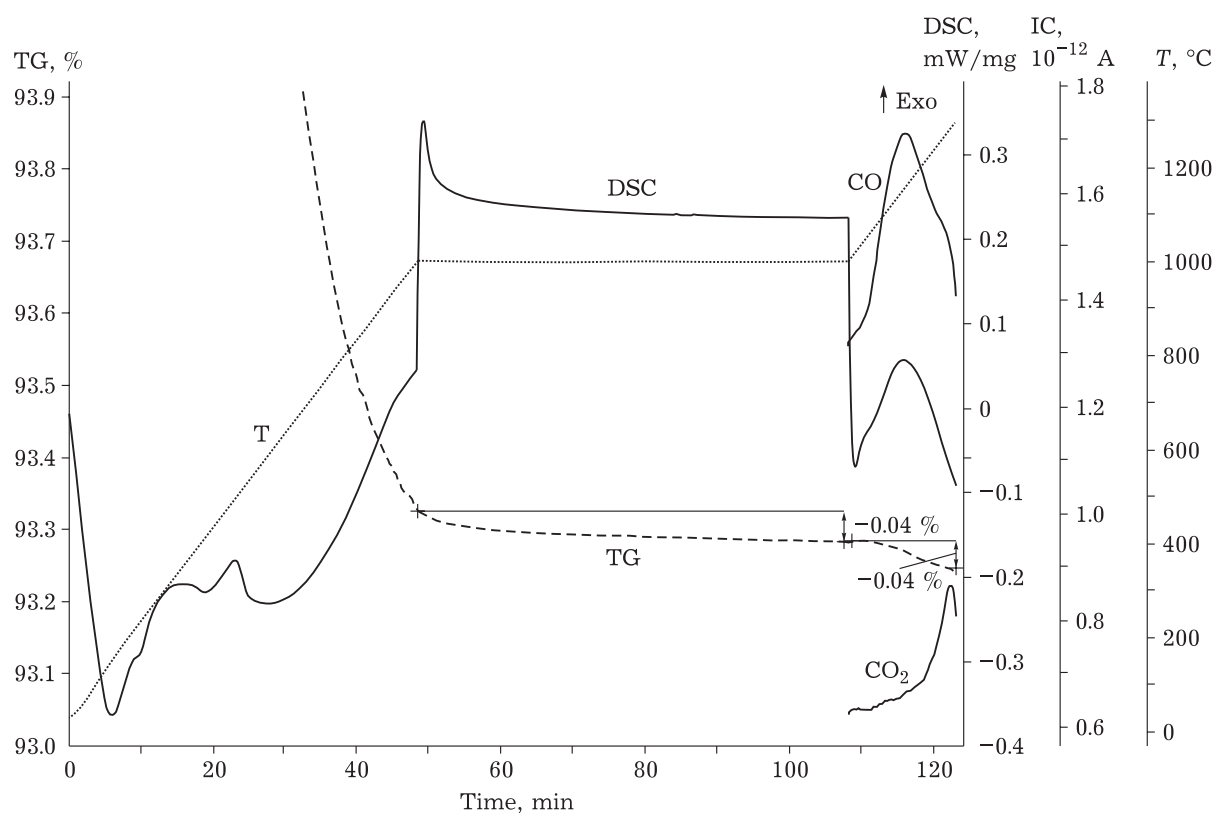


Fig. 3. Thermal and mass spectrometric analysis of the tablet pressed from spinel powder treated mechanically in alcohol.

further sintering. Because of this, the molded samples were heated preliminarily for 1 h at a temperature of 1000 °C in the air to burn the remaining organic molecules, and then vacuum sintering at 1300 °C was carried out. The effect turned out to be insignificant from the viewpoint of an increase in density.

Figure 3 shows the data on the combined thermal and mass spectrometric analysis in the flow of Ar + 20 % O₂ of the molded sample for which isothermal exposure was carried out for 1 h at 1000 °C, and then the temperature was increased to 1300 °C. In spite of the exposure, a clear exothermal effect with the simultaneous release of small amounts of CO and CO₂ is observed, which means that the oxidation of organic molecules occurs. The fact that complete oxidation did not take part even at 1000 °C may be the evidence of the absence of oxygen access to residual organic molecules. Thus, disaggregation of MgAl₂O₄ nanopowder possessing plasticity results in the formation of gas-tight aggregates containing residual alkoxy groups inside. The access of oxygen to the products of carbonization of organic molecules and their removal are provided only after the achievement of substantial mobility of crystal lattice during active shrinkage of the material. Because of this, different methods of disintegration are to be developed for vacuum sintering of powders synthesized mechanically under the described conditions.

CONCLUSION

Mechanochemical treatment of the stoichiometric mixture of aluminium and magnesium hydroxides in the planetary mill at the acceleration of 20g was applied to synthesize precursors that are transformed by heating up to 1000 °C into single-phase magnesium aluminate spinel with nano-sized crystallites. Heating in the atmosphere with increased humidity leads to substantially smaller crystallites. Nevertheless, the sintering of materials molded from these powders does not provide densities sufficient for the subsequent HIP. At the same time, additional mechanical treatment of the obtained spinel in alcohol at the acceleration of 10g helps to achieve the ceramic density of 95 % by sintering in the air at 1350 °C, and these ceramics may be subjected to HIP.

Sintering in vacuum leads to substantially lower densities in comparison with sintering in the air, which is due to the residual alkoxy groups

embedded in gas-tight aggregates, which arise during mechanical treatment in alcohol.

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