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The structural-mechanical properties of the formed pastes and the granular aluminum of magnesium, depending on the conditions of preparation

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Introduction

The use of magnesium aluminum as a carrier of catalytic processes is of significant practical interest due to the neutral acid-base characteristics of the surface and high thermal stability. The traditional methods of obtaining aluminum metals aluminum are high-temperature sintering and gel gel technology. We previously published the results of obtaining aluminum metals using hydrothermal processing of the suspension of the thermal activation of Gibbsite in salts solutions with the production of powder material after calcining [1-3], while granular media and catalysts are used in industry.

The purpose of the work is to obtain granules of the carrier based on the aluminum of magnesium with the structure of the spinel with high values of the specific surface, mechanical strength and volume of pores.

Experimental

The granules were obtained by extrusion forming of pastes through a forming head (filter) with a diameter of 4 mm:

- based on gels formed during hydrothermal suspension processing;
- based on powders obtained after drying gels at 200^0 C;
- based on powders obtained by heat treatment of dried gels at 550^0C.

After molding, all granules were thermal treatment at 550^0 C for 4 hours.

The conditions of hydrothermal processing of suspensions and the starting raw materials are described in [1].

The properties of the molding masses were determined using the Anton Paar rotational viscometer with the RH7 (fig.1) rotor and the conical reservoir of the design of P.A. Rebinder.

The structural mechanical characteristics of the obtained masses were determined using a device with a plane-parallel gap, the principle of operation of which is based on a change in tangential displacement in time, Tolstoy's design

In thermal treatment granules, texture characteristics and mechanical strength were determined. Texture characteristics and a mechanical strength were determined for calcined at 550 ⁰C granules.

The value of the plastic strength and other structural and mechanical properties of the nitrate suspension (sample 0, fig.1) cannot be measured because it has a low concentration of magnesium aluminate and is not suitable for extrusion molding.

Figure 1 -The Anton Paar rotational viscometer with the RH7 rotor and the conical reservoir of the design of P.A. Rebinder.

Table 1- Properties of dried gels

№	Source Mg	S_{sp}, BET/ BJH , м²/g	Vpores,sм³/g	Dpores, nм
1	Nitrate	93.968/71.981	0.220	4.303
2	Oxide	198.366/137.464	0.507	3.138

Table 2 – Properties of molding masses of magnesium aluminate obtained from nitrate and magnesium oxide.

№№	Source product	P_k, kPa	$ή_1$, ·10⁻⁹ Pa·s	Pl, ·10⁶ s⁻¹	λ	E·, MPa	Θ, s	E_1, MPa	E_2, MPa	P_m, кPa	$ε_{fl}$, rel.un.. (%)	$ε_{el}$, rel. un.. (%)	$ε_{pl}$, rel. un.. (%)	SMT
1	Gel Oxide	14,0	21,2	0,77	0,34	1,75	12114	2,67	5,11	2,87	0,75 (61,0)	0,39 (31,7)	0,09 (7,3)	0
2	Paste oxide	17,5	197,3	0,09	0,63	72,7	2714	198,6	114,7	4,45	0,010 26,3	0,017 44,7	0,011 28,0	2
3	Nitrate paste	16,3	353,8	0,04	0,38	76,9	4599	125,0	200,0	6,20	0,016 (50,0)	0,010 (31,2)	0,006 (18,8)	0
4	Paste oxide /550⁰C	19,0	203,3	0,1	0,70	89,1	2282	285,7	125,0	14,4	0,007 (21,2)	0,016 48,5)	0,010 (30,3)	2
5	Nitrate paste /550⁰C	19,3	201,5	0,1	0,69	90,3	2231	289,2	131,4	15,3	0,007 (21,8)	0,015 (46,9)	0,010 (31,3)	2

Pk ,кPa- the value of the limiting shear stress
E, Pa – equilibrium module
ή1, Pa·s - the value of the highest plastic viscosity
E1, Pa - modulus of stretch
E2, Pa – modulus of elasticity
Pl, s-1 - the value of plasticity
λ - the value of elasticity
Θ, s - relaxation period
Pm, кPa - plastic strength
εfl, εel, εpl - reduced deformations, proportions of flexibilitic, elastic and plastic deformations
SMT - Structural and mechanical type

All molding pastes obtained from dried or calcined gels had close values of optimal molding moisture of 20-22%.

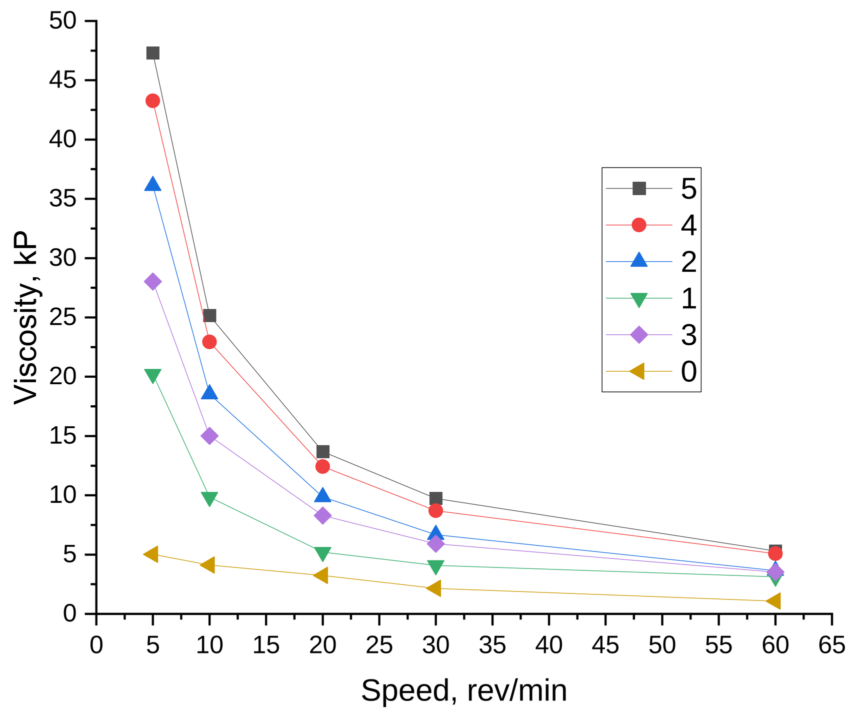

Figure 2 – Dependence of the viscosity value (KP, kiloPuase) from the speed of rotation of the measuring spindle

When analyzing the viscosity values (Fig.1), a significant discrepancy in the indicators can be seen at low speeds of the measuring spindle. It is probably more correct to compare the values of the indicators at a higher speed. Table 4 shows the results of the viscosity value at the rotation speed of the measuring spindle 60 rpm.

Table 4 - Paste viscosity values at the measuring spindle rotation speed of 60 rev/min.

№ №	0	1	2	3	4	5
Visc.., kP	1,08	3,12	3,52	3,65	5,08	5,30

Table 3 - Properties of spinel granules after calcination

№	Source of Mg, Temperature of Gel treatment	S_{sp} BET, м²/g	Strength, MPa	V_{pores}, sм³/g	D_{pores}, nм
1	Oxide, without h/t	200.0	1.5	0.52	3.80
2	Nitrate, 200 ⁰C	150.0	5.0	0.23	3.80
3	Oxide, 200 ⁰C	200.0	3.0	0.52	3.80
4	Nitrate, 550 ⁰C	102.2	3.5	0.22	4.85
5	Oxide, 550 ⁰C	108.2	2.1	0.45	3.10

Conclusion

Magnesium aluminate granules with a spinel structure were obtained by extrusion molding of a mass from a suspension, a powder obtained from it, and a powder spinel obtained by calcining a powder from a suspension at 5500 C. The suspension is obtained by hydrothermal synthesis of the product of centrifugal thermal activation of gibbsite and magnesium compounds (magnesium oxide or nitrate).

The most durable granules (3-5 MPa) with the highest specific surface area (130-200 m2 / g) are obtained from a molding mass prepared from a dried suspension. These masses differ in structural and mechanical properties and belong to different structural and mechanical types (0-th mass of nitrate and 2-th mass of oxide.).

The suspension based on magnesium oxide is suitable for extrusion molding immediately after hydrothermal synthesis. The mass belongs to the 0 structural-mechanical type with a significant predominance of stretch deformations (more than 60%), has the lowest values of plastic strength Pm = 2.87 kPa and viscosity v = 3.12 kP. Granules from this mass are less durable (1.5 Mpa) with the same structural properties (specific surface area, volume and pore diameter) with granules from powder.

The molding mass from the finished spinel has properties similar to ceramic masses: the 2nd structural–mechanical type, the elasticity index λ = 0.60-0.70 and the relaxation period Θ= 2230-2285 s. However, the granules obtained from this mass have a mechanical strength of 2.1-3.5 Mpa and a specific surface area of 100-110 m2/g.

The molding pastes prepared using “nitrate” samples compared to “oxide” samples (from dried or calcined gels) have higher values of viscosity and plastic strength, and calcined granules have higher mechanical strength. The use of magnesium oxide allows to obtain carriers with sufficient strength and higher values of the specific surface area and the volume of pores.

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References:

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