PRODUCTION OF POWDER POROUS MATERIALS FROM MECHANICALLY ACTIVATED ALUMINUM OXIDE

ИЗГОТОВЛЕНИЕ ПОРОШКОВЫХ ПОРИСТЫХ МАТЕРИАЛОВ ИЗ МЕХАНИЧЕСКИ АКТИВИРОВАННОГО ПОРОШКА ОКСИДА АЛЮМИНИЯ

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Abstract *The results of the development of a process for the production of powder porous materials (PPM) based on a granular mechanically activated aluminum oxide powder have been presented. The dynamics of the particle size change of alumina powder depending on the time of mechanical activation by means of a high-energy mill of planetary type has been studied. The development results of permeable materials based on granular mechanically activated oxide ceramic powder have been presented. The developed PPMs are applied for liquid and gas filtration, production of calibration elements for control and measuring devices and equipment.*

KEYWORDS: POWDER POROUS MATERIALS, MECHANICAL ACTIVATION, NANO-SIZED AND ULTRA-DISPERSED POWDERS OF ALUMINUM OXIDE, PROPERTIES

1. Introduction

An important direction of powder metallurgy is the production of porous powder materials (PPM); their efficiency and field of application are determined by the presence of an interconnected pore system. Modern methods of controlling the pore structure in a wide range provide such properties as permeability for liquids and gases, the ability to delay various inclusions, to muffle noise, etc.

In most cases of practical application strict requirements are imposed to porous powder materials on the entire range of properties, primarily to the fineness of filtration and pass-through function [1]. The work quality of the PPM is determined mainly by the fineness of the filtration, which depends on the size of the pores – the smaller they are, the higher the fineness of the filtration. At the same time, the smaller the particles size of the powder from which the filter material is made, the greater the probability of obtaining the material practically impermeable [1]. PPM production technology includes traditional for powder metallurgy operations of charge preparation, molding (with and without pressure) and sintering. The properties of PPM depend on the properties of the initial powders and the technological process for their production. Metal and ceramic powders with particles of various shapes and sizes from nanometers to millimeters are used for their production. Porous materials are produced from powders of bronze, corrosion-resistant steel, titanium, aluminum. Powders of oxides, carbides, silicides and other high-melting compounds are widely used for PPM production.

One of the important directions of porous powder materials is the development of nanomaterials and nanotechnologies. The creation of devices based on oxide ceramics for the filtration of liquids, gases and other purposes is of great interest for research and finds a wide application in various production fields [2-5].

The objective of this work is to develop a process for the production of powder porous materials based on a granular mechanically activated alumina powder.

2. Results and discussions

Mechanical destruction of the powder was carried out in the delivery condition using a high-energy mill of the planetary type "Pulverisette 6" Fritsch (Germany) in order to produce a permeable alumina-based material. When treating powders in high-energy mills, mechanical energy is transferred to the treated substance. As a result, dispergation by milling (particle size reduction) occurs (activation) with a formation of a new surface of particles with a much larger total area per unit mass. The main technological parameters of the mechanical activation of powders affecting the properties of nanosize and ultrafine ceramic powders are the number of rotations of the planetary disk and the treatment time. Mechanical activation of alumina powder was carried out for 10-30 min at the speed of rotation of the planetary disc 550 min^{-1}. The surface morphology of the experimental powder samples was examined on a high-resolution scanning electron microscope "Mira" of "Tescan" company (Czech Republic). The research equipment is shown in Figure 1.

Fig. 1 *Scanning electron microscope of high resolution "Mira-3" of "Tescan" company (Czech Republic)*

Figure 2 shows the images of alumina powder particles in the delivery condition prior to mechanical destruction.

It should be noted that Al_2O_3 powder after milling is characterized by a high tendency to agglomeration and has a complex multilevel structure. Primary ultradispersed and nano-sized particles are combined into agglomerates with the size from 0.5 to several micrometers; it is possible to separate individual nano-sized grains (particles) in their structure.

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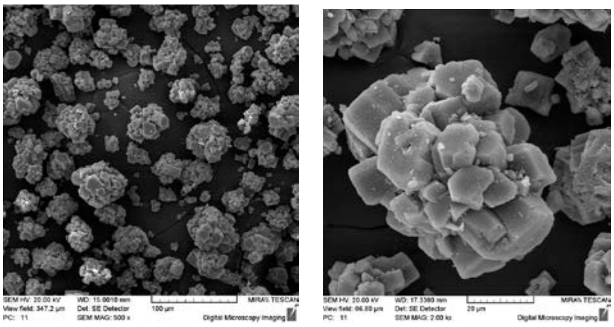
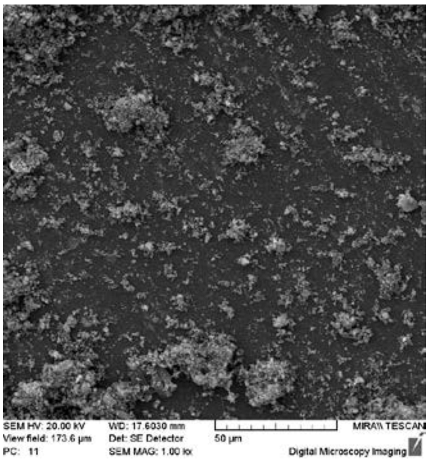
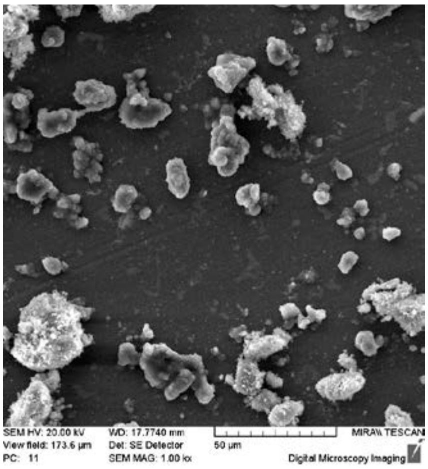


Fig. 2 *Morphology of the surface of alumina powder particles (Al_2O_3), × 500 prior to mechanical destruction*

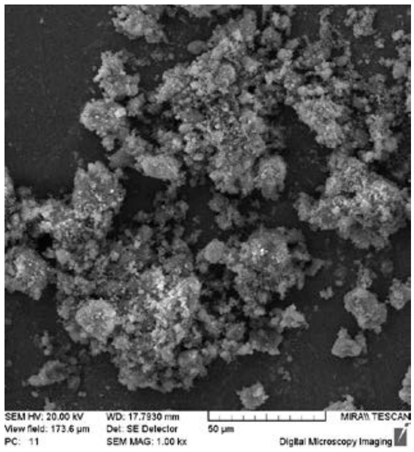
These agglomerates, in turn, are combined into larger (from 10 to 100 µm) secondary aggregates and agglomerates. Considering the difficulty in determining the real particle size of the agglomerate type, and also the fact that it is not possible to completely separate all individual particles during the dispersion process, the size of the primary particles and the maximum (typical) size of the agglomerates was used for comparative assessment visually defined from the SEM micrographs of the powder (Figure 3).



a)



b)



c)

Fig. 3 *Dynamics of the particle size change of aluminum oxide powder depending on the milling time: a) 10 min.; b) 20 min.; c) 30 min., ×1000*

Quantitative stereological analysis of powder samples after mechanical destruction was carried out with the help of the software complex of image processing and analysis "Autoscan". The particle size distribution of the powders is shown in Figure 4.

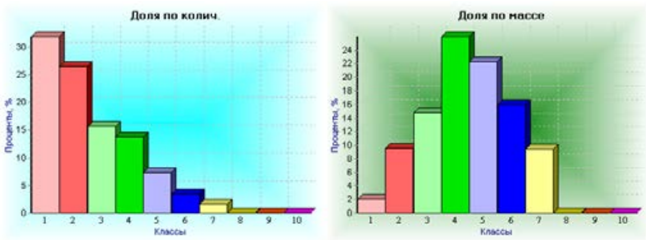


Fig. 4 *Particle size distribution in Al_2O_3 powder after mechanical destruction in a planetary mill for 20 min.: 1 – (0-1,2) µm; 2– (1,2-2,4) µm; 3 – (2,4-3,6) µm; 4 – (3,6-4,8) µm; 5- (4,8-6) µm; 6 – (6-7,2) µm; 7– (7,2-8,4) µm; 8 – (8,4-9,6) µm; 9 – (9,6-10,8) µm; 10 – (10,8-12) µm*

As can be seen from the histogram of the particle size distribution after mechanical activation of the alumina powder, the presence of particles smaller than one micron is 30%.

Mechanically activated aluminum powders were used to produce permeable materials. The technology of PPM production included the following stages: mechanical activation of the powder, charge preparation, pressing, sintering. Mechanical activation of aluminum oxide powder particles was carried out using a high-energy planetary-type mill "Pulverisette 6" of Fritsch company (Germany) for 30 minutes with a rotation speed of 520 min^{-1} of a planetary disk. The particle size of the milled alumina powder was from 50 to 200 nm. The prepared sample weight of aluminum powder was mixed with a 5% solution of polyvinyl alcohol. The obtained mass was dried in a drying cabinet CHBC 4,5.4.5/34-1 at a temperature of 80 °C at constant stirringThe formation of tubular filter materials with a diameter of 12-60 mm and a length of 50-500 mm was carried out by radial isostatic pressing on the isostat ИСПР И1. Sintering of porous materials was carried out in a laboratory oven Netzsch 1700 in air at 1350 °C for one hour.

The samples of permeable materials based on a granular mechanically activated alumina powder are shown in Figure 5.

***Fig. 5** The samples of permeable materials based on a granular mechanically activated alumina powder*

A liquid pollutant (model solution) was used to determine the retention capacity of the developed material. Fertilizer of "HUMO+" was used as a pollutant. The permeable material was placed in a special tooling and sealed. The test liquid pumped by the air source was passed through the permeable material into the tank for filtrate collecting. The filtrate sample was taken and examined for fineness of purification using Laser Diffraction Particle Size Distribution Analyzer Malvern Mastersizer 2000 with an automatic dispersion and delivery module of the HydroS sample. The particle size measuring range of the Laser Diffraction Particle Size Distribution Analyzer Malvern Mastersizer 2000 is 0,02-2000 μm. The fineness of the purification was determined as the maximum particle size found in the filtrate. The device for determining the fineness of purification is shown in Figure 6.

***Fig. 6** Laser Diffraction Particle Size Distribution Analyzer Malvern Mastersizer 2000 with an automatic dispersion and delivery module of the HydroS sample for determining the fineness of purification*

The histogram and diagram of particle size distribution illustrate that the filtrate contains particles ranging in size from 0,02 to 0,3 μm (Figures 7 and 8).

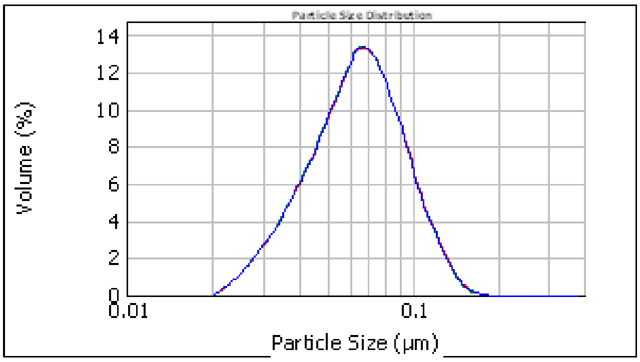


***Fig. 7** The histogram of the particle size distribution contained in the filtrate*

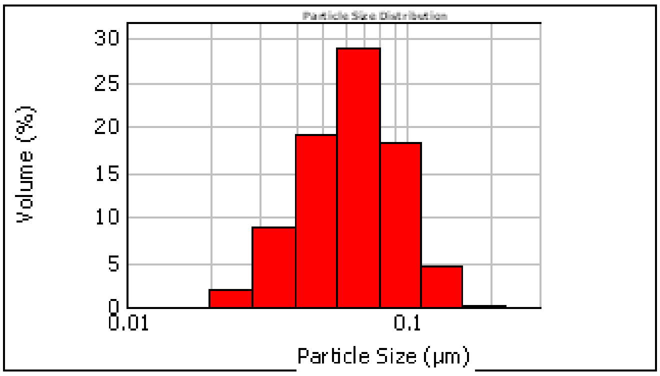


***Fig. 8** Particle size distribution diagram*

The pore size was determined in accordance with GOST 26849-86 by the method of liquid displacement (ethyl alcohol) from the pores; the permeability coefficient is according to GOST 25283-93.
An analysis of the tests has showed that the permeable material based on a mechanically activated alumina powder has an average pore size from 0,55 to 0,8 μm; a permeability coefficient is $(0,55\text{-}0,75)\times10^{-13}$ m^2, and a particle size range contained in the filtrate is 0,02-0,3 μm.

3. Conclusion

The development results of permeable materials based on mechanically activated aluminum oxide powder have been presented. The dynamics of the particle size change of powders depending on the time of milling has been studied. The analysis of the tests showed that the permeable material based on a mechanically activated powder based on aluminum oxide has an average pore size of 0,5-0,9 μm; a permeability coefficient is $(0,55\text{-}0,85)\times10^{-13}$ m^2. Permeable porous materials based on alumina can be produced in the form of a pipe, a disk, a cylinder, depending on the requirements of the design and are used for filtering liquids and gases, for calibration elements of control and measuring devices and equipment.

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