GRAIN ANALYSIS OF HIGHLY DISPERSE POWDERS USING A SCANNING PHOTOSEDIMENTOGRAPH

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A scanning photosedimentograph built by specialists at Ural State Engineering University is described. Results of grain analyses of various refractory materials conducted by means of the device are presented. Data are given for alumina, cement, and quartzite, bauxite, and mullite slips. Experimental dependences of the error of measurement on the concentration of the solid phase are derived. Grain compositions obtained on the photosedimentograph are compared with data obtained on microscreens, and the results are shown to be in satisfactory agreement.

The properties of highly disperse powders depend on the size distribution of the particles. The fraction of surface-active atoms, which determine the reactivity, amounts to tens of percent and grows with the dispersity. As the size of the particles of the refractory powder decreases, the ratio of the contributions of the atoms to the thermodynamic parameters changes quantitatively, and it is therefore important to know the grain composition of the fraction finer than 60 μm because it largely determines the physicochemical properties of the ready product. Practical sedimentation analysis is based on the process of stationary sedimentation of particles in an immobile viscous medium in correspondence with the Stokes law. The equation of stationary sedimentation of a single spherical particle in a viscous medium has the form

\[
\frac{1}{6} \pi d^3 (\rho_p - \rho_m) g = 3 \mu W d,
\]

where \(d\) is the diameter of the particle, \(m\); \(\rho\) is the density of the medium, kg/m³; \(\rho_p\) is the density of the particle, kg/m³; \(\mu\) is the viscosity of the medium, Pa·sec; \(g\) is the free fall acceleration, m/sec²; \(W\) is the sedimentation speed, m/sec.

Equation (1) can be written in terms of criteria as

\[
Re = \frac{W d \rho_p}{\mu},
\]

\[
Ar = \frac{g d^2 (\rho_p - \rho_m) \rho_p}{\mu^2},
\]

where \(Re\) and \(Ar\) are the Reynolds and Archimedes numbers, respectively, which are determined for the particle from the dependences

\[
Re = \frac{W d \rho_p}{\mu},
\]

\[
Ar = \frac{g d^2 (\rho_p - \rho_m) \rho_p}{\mu^2},
\]

The range of application of the Stokes law is limited by \(Re < 1\). Therefore, the maximum diameter of the particles determined by sedimentation analysis should not exceed \(d_{max}\) in the relation

\[
d_{max} \leq \left[ \frac{18 \mu^2}{g (\rho_p - \rho_m) \rho_p} \right]^{\frac{1}{3}}.
\]

When the analysis is conducted in water or alcohol, \(d_{max}\) for the majority of materials with a density ranging from 2000 to 3500 kg/m³ should not exceed 100 μm. For materials that possess a higher density, the test medium should be more viscous, for example, glycerin. The minimum particle size determined by sedimentation analysis is limited by the sedimentation time of the particles, the chosen method of analysis, and the Brownian motion in the liquid medium. For most materials the minimum particle size is 1 μm.

Photosedimentation analysis of the grain composition of powders is based on photometric determination of the degree
of light absorption as a function of time, when the light is transmitted through the powder suspension in the course of the sedimentation. As the light passes through a suspension layer of thickness $\Delta$, it is scattered by the particles. The intensity of the light transmitted through the layer decreases in accordance with the Bouguer–Lambert–Beer law

$$I = I_0 e^{-\tau \Delta},$$  \hspace{1cm} (6)

where $I$ is the intensity of the transmitted light, $I_0$ is the intensity of the incident light, $\Delta$ is the thickness of the suspension layer, and $\tau$ is the turbidity of the suspension.

The Bouguer–Lambert–Beer law holds under the condition $\lambda \ll d$, where $\lambda$ is the wavelength of the light and $d$ is the diameter of a particle. The turbidity of the suspension is rational to the cross-sectional area of the particles and their number in the suspension. If the suspension contains particles of just one size, then

$$\tau = k N s = k_1 N \frac{V}{d} = k_1 \frac{c}{d},$$ \hspace{1cm} (7)

where $N$ is the number of particles per unit volume, $s$ is the cross-sectional area of a particle, $V$ is the volume of a particle, $d$ is its diameter, $c$ is the volume concentration of the particles, $k$ and $k_1$ are rationality factors.

If the suspension contains particles of several classes (for example, $n$ classes), the turbidity is determined by the relation

$$\tau = \sum_{i=1}^{n} \delta \tau_i = k_1 \sum_{i=1}^{n} \frac{c_i}{d_i},$$ \hspace{1cm} (8)

where $\delta \tau_i$ is the fraction of the turbidity created by the $i$-th class, $c_i$ is the volume concentration of the $i$-th class, $d_i$ is the mean size of particles of the $i$-th class, ranging between $d_{i \text{min}}$ and $d_{i \text{max}}$; $d_i$ is determined from the relation

$$d_i = \frac{1}{2} (d_{i \text{min}} + d_{i \text{max}}).$$ \hspace{1cm} (9)

With allowance for Eqs. (6)–(9) the number density of particles of the $i$-th class of coarseness in the suspension can be determined by the formula

$$c_i = \frac{d_i \ln (I_{i \text{min}}/I_{i \text{max}})}{\Delta k_i}.$$ \hspace{1cm} (10)

The grain composition of the powder is determined by the percentage of each class in the solid volume, and therefore the content of the $i$-th class $r_i$ is determined by the formula

$$r_i = 100 \frac{c_i}{\sum_{i=1}^{n} c_i}.$$ \hspace{1cm} (11)

Specialists at Ural State Engineering University have built a scanning photosedimentograph (SPS) for determining the grain composition of finely disperse powders. The use of computer technology to process the signal and a modern element base have made it possible to improve considerably the existing photosedimentation method. A diagram of the device is presented in Fig. 1. Let us consider the main features that distinguish the device from the photosedimentograph described in the monograph of P. A. Kouzov [1]:

1) The gauge is an AL-107A light-emitting diode operating in the infrared wavelength range. This provides noise suppression in the visible spectrum and more highly monochromatic radiation. The receiver is a KFDM low-inertia photodiode. The wavelength of the emitter is 0.9 $\mu$m. Three gauges and three measuring channels are used in order to increase the accuracy of the analysis.

2) Data on the measured photosignal are transmitted in digital form to the serial port of the computer with a 1-sec interval. Any IBM-compatible computer is suitable. After the analysis is finished, the measured data are recorded in a file that contains information on the material and the regime of the experiment, is stored on the hard disk, and can be processed by special software at any time.

3) The carriage with the gauges is equipped with a lifting mechanism. For a certain time the carriage does not move and then it begins to rise and scan the turbidity of the suspension over the height of the cuvette. This shortens the time of the analysis. As a rule, it lasts 10–20 min. The scanning is accompanied by an exact determination of the height of the suspension column, which improves the accuracy of the analysis.

4) Computer processing of the signal, recorded by a special routine, eliminates the effect of the subjective factor and increases substantially the accuracy and reproducibility of the