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A RAPID METHOD FOR DETERMINING THE PARTICLE-SIZE DISTRIBUTION OF MICROPOWDERS

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As the requirements regarding the particle-size distribution of powders, on which the end properties of materials depend to a large extent [1], are becoming more and more severe, there is a need for new, improved instruments for the rapid determination of microparticle sizes. Considerable possibilities are opened up in this field by the use of laser technology for particle-size measurement. A number of industrial models of the laser type are already available, including those made by the Malvern company of Great Britain and Cilas company of France. The principle of operation of these instruments is based on the analysis of Fraunhofer diffraction from several particles located in a measuring zone [1, 2]. The lower limit of diameters of particles being measured is 2 μm. With another variant of instruments of this type (so-called flow-type analyzers), measurements are made of the scattering of monochromatic laser radiation on each particle separately. The flow principle [3-5] makes it possible to establish distribution functions for particles ranging in size from a few hundred angstrom units at a rate of up to 300,000 particles per minute and to grade particles by size [4]. In this respect the method of powder particle-size distribution determination with a flow-type laser analyzer is superior to the existing sedimentation, microscopic, television, and other techniques, which are very time-consuming.

In this article a description is given of a flow-type laser microparticle analyzer, together with the results of particle-size measurements made with it on titanium carbide powders of particle sizes 3-5, 2-3, and 1-2 μm.

A diagrammatic representation of the flow-type laser microparticle analyzer is shown in Fig. 1. A suspension (e.g., in distilled water) of particles being investigated passes under pressure into a hermetic flow-type changer 4 which is transparent to the beam of a helium-neon laser 1. Inside the chamber, the stream of the suspension is hydrodynamically compressed to a diameter commensurable with the size of the particles and focused relative to the laser beam, which at the point of intersection with the stream forms, under the action of two cylindrical lenses 2 and 3, a 10 × 100 μm spot. Light scattered in the direction of the beam from each microparticle is collected by a lens 6 and focused by a photodiode 7. A screen 5 serves to prevent the photodiode from being struck directly by the laser beam. The scatter signals are amplified in 8 and transformed in a unit 9 into 10-sec-long pulses of an amplitude proportional to the area of the incoming pulses, after which they pass to an AI-256 pulse analyzer 10. On the screen of the analyzer there forms a particle scatter distribution curve. The histograms obtained are recorded in a unit 11, their printouts being produced by a digital device 12.

In [3, 4] it is shown that the relationship between the amplitude of a signal proportional to the area of a light pulse of low-angle scatter from a 5-20-μm-diameter spherical particle and the volume of the particle has a proportional character. As a specific example of measurement with the above-described analyzer, Fig. 2 shows the distribution curve of a mixture of single-size calibration spheres of mean diameters 2, 4, and 5 μm. The amplitude of the converted scatter signal, in relative units — numbers of channels of the pulse analyzer 10 (Fig. 1), is marked off along the horizontal axis, and the number of particles n, along the vertical. From these results it follows that proportionality between a signal being measured (number N of an analyzer channel) and the volume of a spherical particle holds even for particles of diameters smaller than 5 μm, since the ratios of the modes of the distributions obtained are proportional to the ratios of the cubes of the corresponding diameters of the measured calibration spheres. Thus, for the particle size range indicated the following formula is valid:

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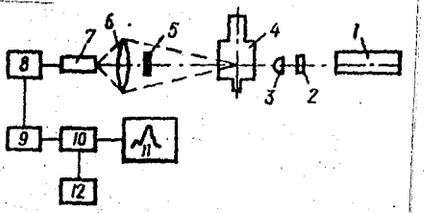

Fig. 1

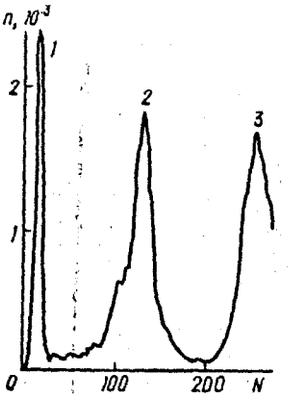

Fig. 2

Fig. 1. Diagrammatic representation of laser microparticle analyzer.

Fig. 2. Curve of distribution of quantity of calibration plastics spheres of diameters 2 (1), 4 (2), and 5 μm (3) over numbers of pulse analyzer channels.

$$N = l\delta^3,$$

where δ is the particle diameter, and l a coefficient of proportionality.

The results presented above confirm also that the use of plastics spheres for the calibration of the analyzer gives satisfactory results, since their distribution obeys the normal law (Fig. 2), and the value of standard deviation of diameters from the mean [6], with allowance for measurement error, does not exceed 7%.

Flow-type laser analyzers can be very successfully employed for the automatic control of the fineness of powders which must meet stringent particle size requirements, such as TiC powders for the manufacture of abrasive pastes. Figure 3 shows distribution curves for titanium carbide powders of particle sizes 3-5, 2-3, and 1-2 μm. The analyzer was calibrated with 5-μm plastic spheres so that to 5-μm-diameter particles there corresponded the 240th channel of the analyzer (Fig. 3). Along the axis of abscissas are marked-off, evenly spaced out analyzer channel numbers and, below them, the equivalent particle diameters δ corresponding to them, found with the expression

$$\delta = \delta_c (N/N_c)^{1/3},$$

where δ_c is the mean diameter of the calibration spheres, and N_c the number of the channel to which the diameter δ_c corresponds after calibration. Thus δ_c = 5 μm and N_c = 240. It should be noted that in further measurements the choice of calibration particles and the matching of their mean diameter with any particular analyzer channel depend on the conditions under which untruncated distributions of micropowders investigated are obtained.

The form of a particle-size distribution function is determined from differential curves, using an equalization method [8]. It was established that the distribution functions (Fig. 3) of particles of titanium carbide powders produced in a ball mill with subsequent centrifugal classification obey the logarithmic normal law. Hence, bearing in mind the large number of levels of amplitude quantization of the signal (256 for the AI-256 analyzer employed), it was assumed that the quantity N, which is in fact a relative amplitude of the signal being measured and a continuous quantity, is also continuous. If the differential function $\varphi_n(\delta)$ of distribution of a quantity of particles n over diameters δ obeys the logarithmic normal law [7] and is expressed by the formula

$$\varphi n(\delta) = \frac{100 \lg e}{\sqrt{2\pi}\delta \lg \sigma} e^{-1/2\left[\frac{\lg(\delta/\delta_{mean})}{\lg \sigma}\right]^2} \quad (1)$$

SOVIET POWDER METALLURGY and METAL CERAMICS Vol. 23, N 11, pp. 825-828, 198

SOVIET POWDER METALLURGY and METAL CERAMICS Vol.23, N 11, pp. 825-828, 1984

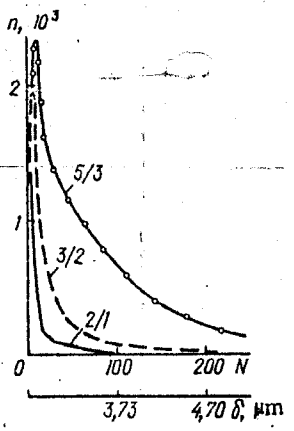

Fig. 3

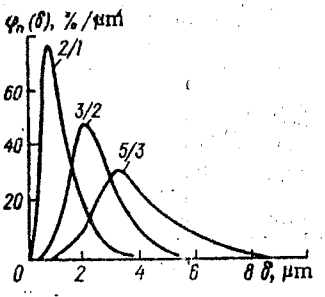

Fig. 4

Fig. 3. Curves of distribution of quantity of TiC powder particles. Along the axis of abscissas are marked off numbers of pulse analyzer channels N and the equivalent sphere diameters δ corresponding to them, and along the axis of ordinates, numbers of particles.

Fig. 4. Differential functions of distribution of numbers of TiC powder particles by size.

(δ_{med} is the median of distribution of over diameters and log σ, the standard deviation of the logarithms of diameters from their mean), then the differential function of distribution $\varphi_n(N)$ of the number of particles n over the parameter N [in fact, the curves of Fig. 3, related to $\varphi_n(\delta)$ by the expression]:

$$\varphi_n(N) = \varphi_n(\delta)\frac{d\delta}{dN}, \tag{2}$$

obeys the same law and is of the form

$$\varphi_n(N) = \frac{100\lg e}{\sqrt{2\pi N}\lg\sigma_N} e^{-1/2\left[\frac{\lg(N/N_{med})}{\lg\sigma_N}\right]^2}, \tag{3}$$

where N_{med} and log σ_N are, respectively, the median and the logarithmic standard deviation of distribution of a quantity of particles over N. The converse of this statement, of course, also holds. Here

$$\lg\sigma = 1/3\lg\sigma_N; \tag{4}$$

$$\delta_{med} = \delta_c(N_{med}/N_c)^{1/3}. \tag{5}$$

It was established that the condition of equalization for the function of being the logarithmic normal law, which follows from the concept of the integral distribution function

$$D(N) = \frac{100}{\sqrt{2\pi}}\int_{-\infty}^{t_N} e^{-\frac{t_N^2}{2}} dt_N = F(t_N), \tag{6}$$

where $F(t_N)$ is a Laplace function [7], and consists in a linear dependence of the argument t_N on the log N in accordance with the equation

$$t_N = \frac{\lg(N/N_{med})}{\lg\sigma_N} \tag{7}$$

was satisfied for all experimentally obtained distributions presented in the form of tables

TABLE 1. Numerical Particle-Size Distribution Characteristics of TiC Powders

Characteristics	Power fraction, μm		
	2/1	3/2	5/3
Mean $\bar{\delta}$, μm	1,27	2,53	3,97
Median δ_{med}, μm	1,08	2,34	3,96
Rms dev. σ_δ, μm	0,79	1,0	1,58
Mode δ_{mod}, μm	0,77	2,02	3,19
Main fraction content determined	0,75	0,5	0,5
in laser apparatus, %	42	41	50
microscopically, %	41	41	49

by the digital printing device of the analyzer. Consequently, the distributions of the titanium carbide powders investigated obeyed the logarithmic normal law with the following logarithmic standard deviations $\log \sigma_N$, determined from the slopes of the corresponding straight lines (7): $\log \sigma_N = 0.75$ for the powder of particle size 1-2 μm and $\log \sigma_N = 0.5$ for the powders of particle sizes 2-3 and 3-5 μm.

Values of N_{med} and $\log \sigma_N$ obtained, using Eqs. (1), (4), and (5), from distributions found by experiment with the aid of the laser analyzer were employed for constructing differential functions of distribution $\varphi_n(\delta)$ of the numbers of particles of the powders investigated by size (Fig. 4) and for determining, using expressions given in [7], their numerical characteristics: mean diameter $\bar{\delta}$, median δ_{med}, rms deviation σ_δ, and mode δ_{mod} (Table 1). In the last two lines of the table are the amounts of the main fractions in the powders, in percent, determined with the laser apparatus and by microscopic analysis. The results obtained by the two methods match, which is evidence that the measurements were correct. It should be noted also that good agreement was obtained between the numerical values of the amounts of any fraction in each powder calculated with Eq. (5) and those found on the basis of experimental results of summation of the number of particles over the channels in the range selected and subsequent division of the sum by the total number of measured particles. This, too, confirms that the distributions of the powders obeyed the logarithmic normal law.

CONCLUSIONS

Use of a flow-type laser analyzer in an investigation of the particle size distribution of a powder markedly accelerates analysis and enables the latter to be performed automatically, with the recording of numerical characteristics and forms of distribution functions for particles ranging in size from a few hundreds of angstrom units to some tens of micrometers. It is shown that the distribution functions of the particles of titanium carbide powders produced by comminution in a ball mill and subsequent liquid centrifugal classification into 3-5-, 2-3-, and 1-3-μm fractions obey the logarithmic normal law. The numerical characteristics of these distributions are given.

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