

THEORY, MANUFACTURING TECHNOLOGY, AND PROPERTIES OF POWDERS AND FIBERS

HYDRODYNAMIC PARTICLE-SIZE CLASSIFICATION OF ALUMINUM ALLOY POWDERS

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Powder fractionation is an essential part of powder metallurgy processes. However, powder particles smaller than 40 μm can hardly be classified. Meshes with openings smaller than 40 μm are not used in conventional sieving because of very low effectiveness since powder is suspended above the sieve as a result of vibration, which makes it difficult for the particles to pass through the mesh. Powders with particles smaller than 40 μm can be fractionated by hydroclassification. The hydroclassification of aluminum alloy powders and distribution of powder particle sizes are studied, and efficient hydroclassification methods are developed. Water-atomized aluminum powders and alloys have not been produced until recently because of the explosion hazard as hydrogen releases when aluminum interacts with water. A method is proposed for the hydroclassification and, thus, fractionation of powders over a wide range of particle sizes, including those smaller than 40 μm , beginning with 0–10 μm .

Keywords: powder fraction, hydrocyclone, inhibitor, grain-size composition, aluminum alloys.

INTRODUCTION

The disintegration of a material (for example, melt atomization) usually results in powders whose grain-size composition is only close to what is needed. Hence, the starting powder is fractionated, the fractions are dosed in a ratio needed and then mixed.

Sieving (screening, bolting, sifting), air classification, and hydrodynamic classification (hydroclassification) are the most popular methods in powder metallurgy used to fractionate powders.

Sieves with openings smaller than 40 μm are not commonly used in powder production because of very low efficiency. The clear area of a 400-mesh (37.5 μm) sieve with \varnothing 25 μm wire is just 35% [1]. In addition, powder particles cannot pass through openings smaller than 40 μm since they are suspended above the sieve because of vibration.

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Powders with particles smaller than 40 μm may be fractionated in air and hydraulic classifiers. However, if active metal and alloy powders whose suspensions are explosive are to be air-classified, special explosion-proof means need to be used, but the cost substantially increases. By their effectiveness and accuracy of grading, hydraulic classifiers are superior to air classifiers [2]. They are especially suitable for wet processes, including the water atomization of metal powders. However, hydraulic classification is currently used in the production of iron powders. Suspensions are usually dehydrated and the resulting powders are dried and graded using vibratory sieves.

The objective of the paper is to examine the hydroclassification and grading of aluminum-based alloy powders and develop efficient hydroclassification methods.

Water-atomized aluminum powders and alloys have not been produced until recently because of the rapid oxidation of their surface in water and explosion hazard from hydrogen gas generation. The issue of explosion safety and oxidation protection of water-atomized aluminum powders was resolved at the Institute for Problems of Materials Science (National Academy of Sciences of Ukraine) [3–6]. This enables the commercial application of this powder production and hydroclassification process and will permit fractionation of powders over a wide range, including size fractions smaller than 40 μm .

EXPERIMENTAL PROCEDURE

To fractionate a water suspension, we selected a hydrocyclone (which is a simple device without moving parts) for grading finely ground materials in the centrifugal field induced by the rotating suspension (Fig. 1).

The powders are produced as follows. The charge prepared using master alloys and containing all alloying elements is molten in an induction furnace. The melt is poured into a hearth and then flows out through a calibration orifice in the bottom. The gravitational metal jet is dispersed with high-pressure jets of preliminary prepared water (its temperature and content of inhibitor and stabilizer being monitored) in a jet chamber. The water suspension with sprayed powder formed in the jet chamber in melt atomization is fed into a sedimentation tank, continuously pumped out from it, and supplied to a hydrocyclone to be graded (Fig. 2). The suspension with coarse particle fractions is returned to the sedimentation tank, the powder settles down in its lower section, and is mechanically dehydrated by filtration under vacuum. The suspension with fine fractions is settled down in a thickener or particles are separated on a filter. After dehydration, the wet powders are vacuum-dried.

To optimize the sedimentation and dehydration, we have examined the kinetics of gas release when atomized powders from aluminum-based alloys doped with scandium, titanium, and zirconium interact with water.

The kinetics of gas emission resulting from the interaction of aluminum alloy with water was examined depending on the suspension parameters (pH, temperature). The rate of gas emission, which characterizes the oxidation of powders in water, is represented by q , $\text{ml}/(\text{kg} \cdot \text{h})$, and is determined as the amount of gas per 1 ml formed within 1 h when 1 kg of powder interacts with water at room temperature. It was assumed that particles 0.25 μm at the most settled down in the thickener for 32 h. The suspension should have such parameters (inhibitor composition, hydrogen index) that particles settle down for a shorter period of time than the induction period of powder oxidation in water.

To examine the kinetics of gas emission, we used an original system that applies a force to balance the pressure of the gas accumulated under the measuring dome [7]. The experimental procedure allows for the peculiarities of the alloy powders used and is based on the following principles. The parameters of the measuring dome are determined from the expected rate of gas emission and the error ($\pm 1\%$) admissible in the measurement of the gas volume. Powder samples are taken from the sedimentation tank of the spraying apparatus in an amount sufficient to examine the chemistry and morphology of particles. If several samples cannot be measured in parallel, they are stored at a temperature no higher than 5°C.

The weight distribution of particles larger than 40 μm was studied by dry sieving according to ISO 4497. The grain-size composition of the minus material was determined using a laser granulometer and then checked by finding the diameters of particles in microphotographs taken with a scanning electron microscope (SEM). Therefore, the results were selectively checked with a laser granulometer. This was needed because particles of superfine powders prepared for laser granulometry may be nondiscretely distributed, and the device may regard interacting particles as one.

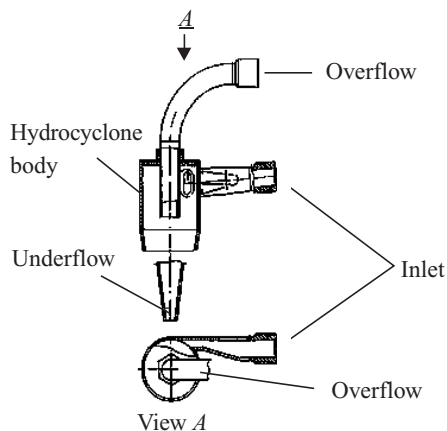


Fig. 1. General view of hydrocyclone

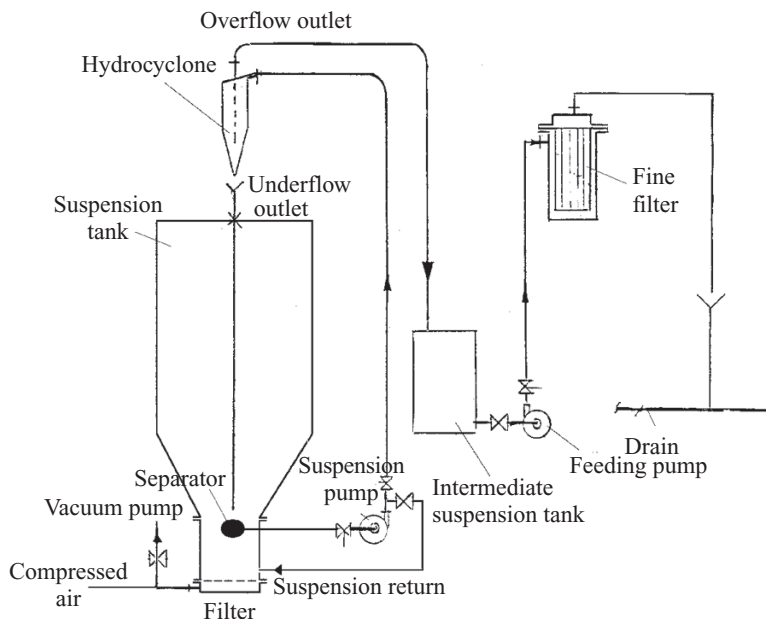


Fig. 2. System for hydroclassification of water-atomized powders

The surface morphology and shape of powder particles were examined with a scanning electron microscope.

EXPERIMENTAL RESULTS

We examined the macroscopic kinetics of powders interacting with water solutions of dispersion inhibitors (weak electrolyte group) and stabilizers such as protective colloids and their mixture if pH of water varies from 3.0 to 6.0. Samples of wet powder are taken directly from the vacuum filter chamber once the pulp has been dehydrated (Fig. 2), which is different from the known methods that use commercial powders or experimental samples of dried powders with a dense oxide film over particles to examine the kinetics of gas emission in water [8]. The compositions of our alloys are summarized in Table 1 and the rates of gas emission at room temperature in Table 2.

If there is no dispersion stabilizer over a pH range between 5.0 and 6.0, the rate of powder–water reaction increases in 6 h, which results in more intensive gas emission up to 20–40 ml of gas per 1 kg of dry powder per hour. With these pH values, the reaction becomes more and more intensive with longer interaction of all samples with water. When hydrogen generation becomes avalanche-like, as is the case for $\text{Al}_{63}\text{Cu}_{25}\text{Fe}_{12}$ powder in water after 168 h, the powder is self-heated to 100°C and is completely oxidized. The nature of powder–water interaction fundamentally changes in water solution of corrosion inhibitor, which decreases pH to 3.0–4.0, and after addition of dispersion stabilizer (0.007 wt.% naphthenic soap) into the solution. The rate of gas emission did not reach 5 ml/(kg · h) at pH 4.0 and was no higher than 1 ml/(kg · h) at pH 3.0 during observation of the reaction rate. Hence, this time was not enough for the induction period of the powder–water reaction to end.

TABLE 1. Chemical and Grain-Size Composition of Powders from Alloys Selected for Studying Gas Generation Kinetics

Alloy No.	Content of elements, wt. %	Content of particles (wt. %) with size (μm)						
		+200	–200+160	–160+100	–100+63	–63+50	–50+40	–40
1	$\text{Al}_{91.35}\text{Zn}_{4.2}\text{Mg}_{3.5}\text{Cu}_{0.6}\text{Zr}_{0.05}\text{Sc}_{0.2}\text{Ti}_{0.1}$	10.3	5.8	20.2	17.6	10.2	16.1	19.8
2	$\text{Al}_{63}\text{Cu}_{25}\text{Fe}_{12}$	6.9	6.0	20.4	35.5	11.3	14.2	4.7
3	$\text{Al}_{94}\text{Fe}_{2.5}\text{Cr}_{2.5}\text{Ti}_{0.7}\text{Zr}_{0.3}$	20.8	8.9	19.2	19.3	9.3	12.5	10.0

TABLE 2. Gas Generation Rate q , ml/(kg · h) in Interaction of Aluminum Alloy Powders with Water

Duration, h	$Al_{91.35}Zn_{4.2}Mg_{3.5}Cu_{0.6}Zr_{0.05}Sc_{0.2}Ti_{0.1}$				$Al_{63}Cu_{25}Fe_{12}$			
	pH 6.0	pH 5.0	pH 4.0	pH 3.0	pH 6.0		pH 5.0	
					A*	A**	A*	A**
1	10	5	1	0	8	1	4	1
6	40	20	3	1	25	1	20	1
12	54	40	4	1	39	1	23	1
24	65	52	4	1	50	1	25	1
30	80	58	4	1	61	1	28	1
36	90	75	4	1	68	1	38	1
48	104	100	4	1	73	1	40	1
108	250	150	5	1	180	1	90	1
168	400	180	5	1	4800	1	600	1

Duration, h	$Al_{63}Cu_{25}Fe_{12}$		$Al_{94}Fe_{2.5}Cr_{2.5}Ti_{0.7}Zr_{0.3}$			
	pH 4.0	pH 3.0	pH 6.0	pH 5.0	pH 4.0	pH 3.0
1	3	1	2	1	1	0
6	3	1	6	2	1	0
12	3	1	10	6	1	0
24	3	1	10	6	2	0
30	3	1	18	8	2	1
36	3	1	25	12	2	1
48	3	1	85	30	2	1
108	3	1	210	80	2	1
168	3	1	420	98	2	1

A* is gas generation rate in tests without dispersion stabilizer; A** is gas generation rate with dispersion stabilizer in water (0.007 wt.% naphthenic soap).

The experimental results allowed us to optimize the composition of water solution of corrosion inhibitor for melt dispersion. The powder–water interaction (1 ml/(kg · h)) is prevented in weak electrolyte solution with pH 3.0 containing no more than 0.007 wt.% naphthenic soap. Note that 1 ml/(kg · h) is insignificant and this reaction path may lead only to two or three alumina monolayers formed for 30–40 h. Hence, holding the suspension in the thickener for 32 h, which is needed to concentrate material with particles smaller than 20 μ m, will not intensify powder oxidation.

The metal melt was atomized with water with a temperature of 6°C and pH 3.0 under 15 MPa. The preset pH value was maintained by the automatic control system. A needed powder fraction was separated in a cyclone with \varnothing 50 mm cylindrical section by controlling the suspension feeding rate and level of the mouth of the overflow outlet in the hydrocyclone (Fig. 1). To optimize hydroclassification (to separate fractions smaller than 20 μ m), the effectiveness of suspension fractionation under four conditions differed in the suspension feeding rate and the level of the mouth of the overflow outlet was examined (Fig. 1, Table 3). The suspension with a separated fine fraction was either let settle down in the thickener or filtered. The concentrate, just as the filter sediment, was dried.

Data on grain-size composition of powders from different aluminum alloys obtained by dry sieving, in accordance with ISO 4497, are summarized in Table 4. As seen, the grain-size composition of all powders is characterized by high content of the fraction smaller than 40 μ m. The powder from $Al_{94}Fe_{2.0}Cr_{2.0}Ti_{2.0}$ alloy containing the largest amount of particles smaller than 40 μ m was selected for further study of hydroclassification.

TABLE 3. Hydroclassification Modes for Water-Atomized Powders

Parameter	Mode			
	A	B	C	D
Inlet pressure, MPa	0.012	0.02	0.032	0.04
Suspension feeding rate, m/sec	1.065	1.39	1.710	1.91
Level of the mouth of the overflow outlet	At the level of the lower edge of the inlet		10 mm below the lower edge of the inlet	

TABLE 4. Grain-Size Composition (wt.%) of Water-Atomized Powders Based on Dry Sieving

Alloy	Particle fraction, μm						
	+200	-200+160	-160+100	-100+63	-63+50	-50+40	-40
$\text{Al}_{94}\text{Fe}_{2.0}\text{Cr}_{2.0}\text{Ti}_{2.0}$	10.6	2.3	5.8	10.9	6.8	15.3	48.3
$\text{Al}_{94}\text{Fe}_{4.1}\text{Cr}_{1.3}\text{Zr}_{0.6}$	4.3	2.8	8.1	16.0	6.7	21.9	40.2
$\text{Al}_{94}\text{Fe}_{2.5}\text{Cr}_{2.5}\text{Ti}_{0.7}\text{Zr}_{0.3}$	8.5	4.6	1.2	20.6	6.9	19.4	38.8
$\text{Al}_{94}\text{Fe}_{4.1}\text{Cr}_{1.3}\text{Ti}_{0.47}\text{Zr}_{0.13}$	3.7	2.6	7.9	16.3	5.8	19.0	44.7

TABLE 5. Grain-Size Composition of Powder Fractions Separated with Hydroclassifier

Particle fractions, μm	Content of fractions, wt.%, separated in different modes							
	A		B		C		D	
	Lsr	Mnl	Lsr	Mnl	Lsr	Mnl	Lsr	Mnl
30-25	12.3	14.4	12.6	0.0	3.4	0.0	7.3	0.0
25-20	16.4	15.7	23.5	11.2	14.0	0.0	15.8	0.0
20-15	24.1	18.5	16.8	21.0	23.3	26.8	24.6	14.1
15-10	30.8	36.4	24.4	42.1	36.2	39.1	33.4	38.5
10-5	12.0	14.0	17.0	25.2	17.4	33.8	14.0	46.6
<5	4.4	1.0	5.7	0.5	5.7	0.3	4.9	0.8

Table 5 summarizes data on the grain-size composition of powder fractions separated in hydroclassification, which was analyzed with a laser granulometer (Lsr) and based on diameters of particles (Mnl) determined by measuring the areas of the horizontal projections of particles in microphotographs taken with a scanning electron microscope.

Comparing the results obtained with a laser granulometer and determined “manually” shows that the former makes the grain-size composition coarser over a range of particle sizes between 5 and 15 μm , i.e., it underestimates the content of fine fractions and overestimates the content of coarse fractions, thus showing the presence of coarse particles that do not even exist. This confirms the assumption on potential errors of laser granulometry in determining the content of fine particles. At the same time, the “manual” method shows lower content of particles smaller than 5 μm as compared with laser granulometry, which may be attributed to inadequate magnification of microphotographs.

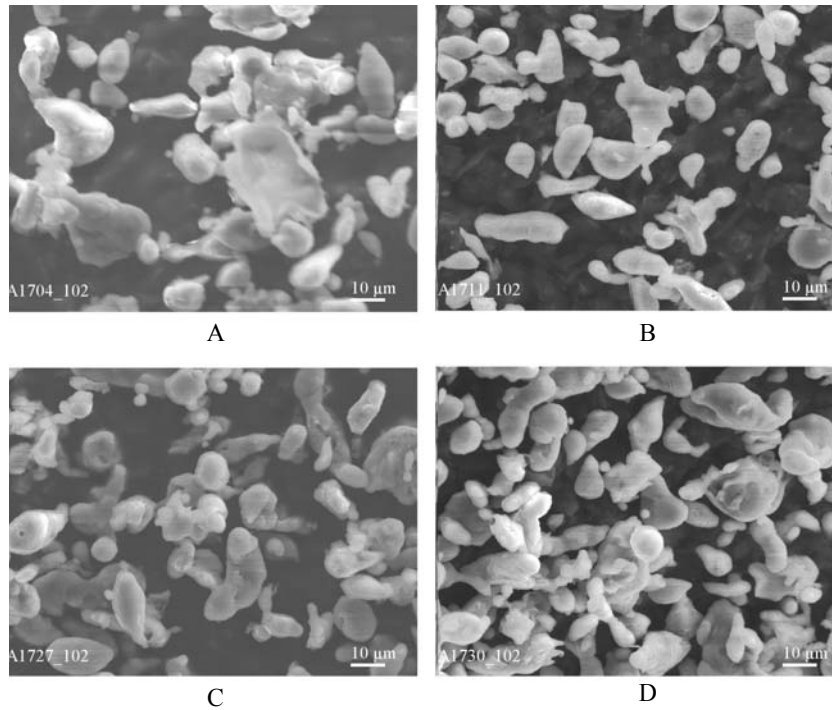


Fig. 3. Microphotographs of fine powder fractions separated using the hydrocyclone in modes A, B, C, and D

Typical microphotographs of fine powder fractions separated in different hydroclassification modes are shown in Fig. 3. There are particles larger than 20 μm (30–25 μm and 25–20 μm fractions) in mode A (Table 5). There are no particles larger than 20 μm in separated powder fractions in modes C and D.

The applicability of lognormal distribution of particles in ground material by their diameters, which was earlier theoretically proved by Academician Kolmogorov for materials mechanically ground for a long time [9], has been shown for water-atomized [10] and gas-atomized [11] powders.

Let us ascertain whether this distribution is valid for powder fractions smaller than 40 μm separated by hydroclassification from one powder batch.

Figure 4 shows the lognormal distribution of calculated (“manually” determined) size fractions of aluminum powder separated from one batch in mode C (Table 5) and fractions of $Al_{94}Fe_{2.0}Cr_{2.0}Ti_{2.0}$ alloy powder separated from one batch in mode D.

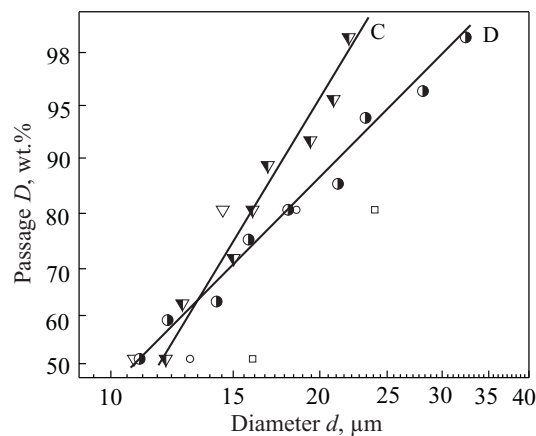


Fig. 4. Diameter distribution of particle masses

TABLE 6. Median Diameter d_{50} and Standard (Root-Mean-Square) Deviation σ of Lognormal Distribution of Water-Atomized Aluminum and $\text{Al}_{94}\text{Fe}_{2.0}\text{Cr}_{2.0}\text{Ti}_{2.0}$ Alloy Powders

Material	Particle mass distribution		Particle surface distribution	
	d_{50}	σ	d_{50}	σ
Aluminum	12	1.33	7.3	1.41
$\text{Al}_{94}\text{Fe}_{2.0}\text{Cr}_{2.0}\text{Ti}_{2.0}$	11	1.65	4.7	1.59

We see that the size distribution of particle masses is almost linear. Therefore, the density of the diameter distribution of particle masses may be represented as

$$\varphi(d) = \frac{100}{\sqrt{2\pi}} \frac{\lg e}{d \lg \sigma} \exp\left(-t^2/2\right), \quad (1)$$

where $\lg e = 0.4343$; d is the particle diameter; σ is the standard (root-mean-square) deviation of parameters from a tabulated function $f(t)$; the minus material constitutes 84.1% at $t = +1$ and 15.9% at $t = -1$ [9].

Hence, the distribution is characterized by two parameters: mean median diameter of particles d_{50} and σ :

$$\sigma = \frac{d_{84.1}}{d_{50}} = \frac{d_{50}}{d_{15.9}}, \quad (2)$$

where d_{50} is the median diameter (the particles with larger diameters weigh 50% of the total weight, the other 50% falling on the particles with smaller diameters).

The root-mean-square (standard) deviation of parameters from their mean value is characterized by the slope of distribution lines. The size distribution of particle surface is as follows:

$$D_s(d) = F(t + \ln \sigma), \quad (3)$$

where $F(t + \ln \sigma)$ is normalized distribution function with argument $t + \ln \sigma$; t is expressed in terms of d as

$$t = (\lg d - \lg d_m) / \lg \sigma. \quad (4)$$

Table 6 summarizes d_{50} and σ of aluminum and $\text{Al}_{94}\text{Fe}_{2.0}\text{Cr}_{2.0}\text{Ti}_{2.0}$ alloy powders obtained under a water pressure of 15 MPa, liquid-to-solid ratio (water and powder weights) $l/s = 15-20$, optimal content of the inhibitor in water, optimal temperature of the suspension in tank, and optimal pH.

Hence, lognormal distribution is valid for powder fractions smaller than 40 μm separated from one powder batch; the grain-size composition of these fractions can also be characterized by two parameters: d_{50} and σ .

The most efficient parameters for separating powder fractions $<20 \mu\text{m}$ in a $\varnothing 50 \text{ mm}$ hydrocyclone are hydraulic heads $p_s = 0.02$ and 0.032 MPa and feeding rates of 1.39 and 1.71 m/sec , respectively.

The system for the hydroclassification of powders permits separating fractions between 0 and 40 μm beginning with 0–10 μm . Dried fractions coarser than 40 μm are graded using vibratory sieves.

CONCLUSIONS

The fractionation of water-atomized aluminum alloy powders for particles smaller than 40 μm in hydraulic classifiers has not been used in powder metallurgy because powders are oxidized in long interaction with water and because of an explosion hazard as hydrogen releases in intensive aluminum-water interaction.

Use of water solutions of dispersion inhibitors (weak electrolytes) and stabilizers (protective colloids) protects the surface of water-atomized powders of aluminum and its alloys against oxidation and prevents gas

generation, thus permitting the hydroclassification and fractionation of powders over a wide range, including fractions smaller than 40 μm .

A weak electrolyte solution with pH 3.0–4.0 or a solution containing 0.007 wt.% naphthenic soap, with controlled gas release being no more than 1–3 ml/(kg · h), is optimal for atomizing melts of aluminum alloy powders. The concentration process lasting for up to 32 h to settle down particles 0.25 μm at the most hardly leads to hydrogen generation or increase in the level of oxidation.

To separate powder fractions <20 μm in a \varnothing 50 mm hydrocyclone, a feeding rate of 1.39 and 1.71 m/sec proved to be most efficient. The hydroclassification of powders permits separating different fractions between 0 and 40 μm , beginning with 0–10 μm .

Lognormal distribution is valid for water-atomized powder fractions smaller than 40 μm separated from one powder batch and, hence, the grain-size composition of these fractions can also be characterized by two parameters: d_{50} and σ .

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REFERENCES

1. T. Allen, "Sampling and classification of powders," in: *ASM Handbook, Vol. 7*, ASM International Publishers, (1998), pp. 206–221.
2. M. D. Barskii, *Powder Fractionation* [in Russian], Metallurgiya, Moscow (1980).
3. O. D. Neikov, "Water atomized powder technologies for advanced aluminum alloy production," in: *Proc. 2000 Powder Metallurgy World Congress*, Jap. Society of Powder Metallurgy, Kyoto (2000), pp. 464–466.
4. O. D. Neikov, A. V. Krajnikov, Yu. Milman, et al., "Advanced PM aluminum alloys produced by new rapid solidification technology," in: *Proc. PM'2004 World Congress, Vol. 1*, European Powder Metallurgy Association, Shrewsbury, UK (2004), pp. 237–242.
5. O. D. Neikov, Yu. V. Milman, A. I. Sirko, et al., "Elevated temperature aluminum alloys produced by water atomization. Water atomized aluminum alloy powders," *Mat. Sci. Eng. A*, **477**, 80–85 (2008).
6. A Method to Make Half-Finished Products from Aluminum Alloys [in Russian], *Patent of Ukraine No. 66588*, Byul. No. 9, September 15 (2005).
7. O. V. Efremov, O. D. Neikov, P. B. Rabin, and M. F. Salamatina, "Microencapsulation of metal powders for preventing detonation and dust in their production and conversion. III. The kinetics of gas evolution," *Powder Metall. Met. Ceram.*, **30**, No. 10, 892–895 (1991).
8. A. K. Lokenbakh, N. A. Zaporina, and L. K. Lepinu, "Phase compositions and structure of oxide films on fine aluminum powder particles," *Tr. Latv. AN, Ser. Khimiya*, No. 1, 45 (1981).
9. P. A. Kouzov, *Fundamentals of the Particle-Size Analysis of Industrial Dusts and Ground Materials* [in Russian], Khimiya, Leningrad (1974).
10. J. J. Dunkley, "Atomization," in: *ASM Handbook, Vol. 7*, ASM International Publishers (1998), pp. 35–52.
11. A. Lawley, *Atomization*, Metal Powder Industries Federation Publishers, Princeton (2003).