

Development of a phenomenological model of air-centrifugal classification of Kovar precision alloy powders

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Abstract: The urgent task is to develop models for the effects of classifier operating modes on the granulometric composition of fine powder fractions separated by the air-centrifugal method, because the use of sieve classification is not effective for the dust fraction of powders with a size of less than 40 μm, which has great potential for use outside of classical additive technologies. In this study, a small amount of Kovar precision alloy powder was classified using air-centrifugal technology for powder injection molding (PIM) applications. The aim of the research was to develop a model that would allow us to select the optimal conditions for air-centrifugal classification in order to obtain a powder with a specific grain size distribution. The methods of laser diffraction and scanning electron microscopy were used to determine the granulometric composition and morphology of the powder. A method of isoconversion analysis of granulometric composition is described, which allows one to obtain model-free (i.e. without hypotheses about the type of kinetic model) single-factor dependencies of the influence of the frequency of air-centrifugal classification on the granulometric composition of the final fraction of the obtained powder. The practical significance of this research lies in the potential for producing powder fractions with a specific average particle size using any combination of a powder with an initial granulometric composition and an air-centrifugal classifier.

Keywords: powders; Kovar; dispersed fillers; particle size distribution; air-centrifugal classification; laser light scattering; powder metallurgy; additive technologies; composite materials.

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Разработка феноменологической модели воздушно-центробежной классификации порошков прецизионного сплава Ковар

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Аннотация: Актуальной задачей является разработка моделей влияния режимов работы классификатора на гранулометрический состав тонких фракций порошка, выделяемых воздушно-центробежным методом, так как использование ситовой классификации не эффективно для пылевой фракции порошков с размером менее 40 мкм, имеющей большой потенциал применения вне классических аддитивных технологий. В рамках данной работы тонкая фракция порошка прецизионного сплава Ковар классифицировалась для использования в технологии инъекционного литья порошков – PIM. Цель работы – построение феноменологических зависимостей, позволяющих подбирать режимы воздушно-центробежной классификации для получения фракции порошка с заданным гранулометрическим составом. Для определения гранулометрического состава и морфологии порошка использовались методы лазерной дифракции и растровой электронной микроскопии. Описана методика изоконверсионного анализа гранулометрического состава, позволяющая модельно-независимо (то есть без гипотез

о виде кинетической модели) получать однофакторные зависимости влияния частоты воздушно-центробежной классификации на гранулометрический состав итоговой фракции получаемого порошка. Практическая значимость данного результата заключается в возможности получения фракций порошка с заданным средним размером частиц для любой комбинации «порошок исходного гранулометрического состава – прибор для воздушно-центробежной классификации».

Ключевые слова: порошки; сплав Ковар; дисперсные наполнители; гранулометрический состав; воздушно-центробежная классификация; лазерное светорассеяние; порошковая металлургия; аддитивные технологии; композиционные материалы.

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1. Introduction

The subject of this study is the relationship between the granulometric composition of fine powder fractions produced during air-centrifugal classification and the operating parameters of the classifier. This study is relevant because sieve classification is ineffective for powders smaller than 40 microns in size, and therefore, air-centrifugal classifiers are required [1–3]. At the same time, there are many applications for laboratory production of powder fractions with specific granulometric compositions, such as additive technologies, dispersed composites, and powder metallurgy. For this study, fractions of precision alloy Kovar powder were classified using air-centrifugal technology for use in PIM (powder injection molding), injection casting, which is detailed in [4–6]. The classification of fine powders can potentially lead to a wider range of applications for powders with different fraction compositions, including fine fractions with a size less than 40 μm , which are not suitable for additive manufacturing. This could increase the overall yield for powder producers [7]. Despite the fact that industrial classification models and powder classification kinetics are well developed, the challenge of producing powders with a specific granulometric composition remains a practical challenge for many laboratories [8–10]. The aim of this study is to develop a set of phenomenological equations for determining the parameters of air-centrifugal classification in order to obtain powder fractions with the desired granulometric distribution in laboratory settings.

2. Materials and Methods

2.1. Starting materials

For the air-centrifugal classification of fine fractions, we used a spheroidized powder of nickel-cobalt iron alloy 29NK (particles size $D < 100 \mu\text{m}$)

produced by RedMetAlloy LLC (Yekaterinburg, Russia) as a starting material.

This alloy, also known as Kovar, belongs to group III of precision alloys according to Russian Standard 10994 due to its strictly defined temperature coefficient of linear expansion (TCLE). The TCLE value of the 29NK alloy is provided by the chemical composition (Table 1). The iron content not specified in Table 1 ranges from 51.1 to 54.5 % by weight in alloy 29NK.

Alloy 29NK is widely used for vacuum soldering of electronic equipment components with glass. This is due to the fact that in a wide temperature range, alloy 29NK has a TCLE close in value to the TCLE of borosilicate glass used in fluorescent lamps, vacuum tubes, metal-glass insulators and metal-ceramic microcircuit housings. At the same time, alloy 29NK has high adhesion to molten glass, due to which it is used to manufacture electrical terminals of electronic devices passing through glass or ceramics, forming a reliable vacuum-tight connection during soldering. Thus, due to TCLE and adhesion, precision nickel-cobalt iron alloy grade 29NK, also known as Kovar, is used to manufacture hermetic connections with glass operating in a wide temperature range [11–13].

Table 1. Chemical composition of 29NK alloy, % by weight

Element	Content	Element	Content
Ni	28.5–29.5	Cu	≤ 0.2
Co	17.0–18.0	Cr	≤ 0.1
Mn	≤ 0.4	C	≤ 0.03
Si	≤ 0.3	S	≤ 0.015
Al	≤ 0.2	P	≤ 0.015

Table 2. Main characteristics of the IG-6UM air-centrifugal classifier

Characteristic	Meaning
Maximum particle size in the source material, μm	No more than 600
The lower boundary of the dimensional separation, μm	3
The maximum volume of a single loading of the initial powder, L	No more than 0.8
Productivity, $\text{kg}\cdot\text{h}^{-1}$	0.5–4.0
Rotation speed of the separation unit, rpm	400–6000

2.2. Equipment and conditions for air-centrifugal classification

For the air-centrifugal classification of the initial powder of the 29NK alloy, an IG-6UM classifier manufactured by NTC INOX LLC (Novosibirsk, Russia) was used. This classifier is designed for dry separation of powder materials into narrow dimensional fractions. The main technical characteristics of the IG-6UM are shown in Table 2.

In this study, the classification of the initial powder of 29NK alloy was conducted at three different frequencies: 7, 10, and 12 Hz. For all three cases, the volume of the initial sample was the same, amounting to 200 mL, and the separation duration was also the same at 60 min for all modes. This approach ensured that the classification process could be completed in time for any selected rotational speed of the separation unit. Therefore, for the chosen sample of initial powder, a phenomenological classification model could be constructed that does not consider the kinetic time factor or intermediate conversion states of granulometric composition.

2.3. Analytical methods

After classification, the granulometric composition of the 29NK alloy powder was determined using laser diffraction according to ISO 13320, with the Microtrac Bluewave device. This method is based on analyzing the scattering of a laser beam by particles in a liquid stream, with a known refractive index. Isopropanol, with n_D of 1.3776, was used as the liquid.

The fine powder fractions obtained from the classification were then analyzed using scanning electron microscopy (SEM). An NT 3200 scanning

electron microscope was used, with an accelerating potential difference of 15 kV, in the back-scattered electron (BSE) mode. Secondary electrons (SE) were used to evaluate the structure of individual particles.

3. Results and Discussion

The results of measuring the granulometric composition of 29NK alloy powder after air-centrifugal classification are graphically shown in Fig. 1. In Fig. 1, the following designations are used for the curves: D_v represents the diameter distribution of spherical particles with an equivalent volume, and D_s represents the diameter distribution of particles with an equivalent surface area. The main parameters for these distributions are presented in Table 1.

Since the fine powder fractions were obtained by air-centrifugal classification, the differential distributions of their sizes observed in Fig. 1 are not truncated, which would be typical for the sieve classification method.

For all classification frequencies in Fig. 1, there is a similarity and proximity of the distribution of the diameters of spherical particles with an equivalent volume D_v to the distribution of the diameters of spherical particles with an equivalent surface area D_s . Thus, the analysis of laser diffraction data allows us to conclude that the powders under study have a shape close to spherical. This shape of the particles is explained by the fact that the studied powder of alloy 29NK was produced by gas atomization.

To confirm the conclusion about the spherical shape of powder particles, as well as to compare the morphological composition of the fine fractions obtained at three different classification frequencies, SEM photographs of powder particles from the fractions obtained at air-centrifugal frequencies of 7, 10, and 12 Hz were obtained using a scanning electron microscope. These photographs are shown in Fig. 2.

The photographs shown in Fig. 2a, 2c, 2e confirm that, regardless of the classification rate, all three fractions of the 29NK alloy powder contain predominantly spherical particles. At the same time, the SEM-images obtained in the secondary electron (SE) mode show – Fig. 2b, 2d, 2f – that the 29NK alloy powder obtained by gas atomization has a not very smooth surface with small satellite particles and visible pores. This is typical for this method of powder production [15–17]. If the PREP method – Plasma Rotating Electrode Process – had been used to produce the powder, the powder would have had a strictly spherical shape without satellites and obvious signs of porosity.

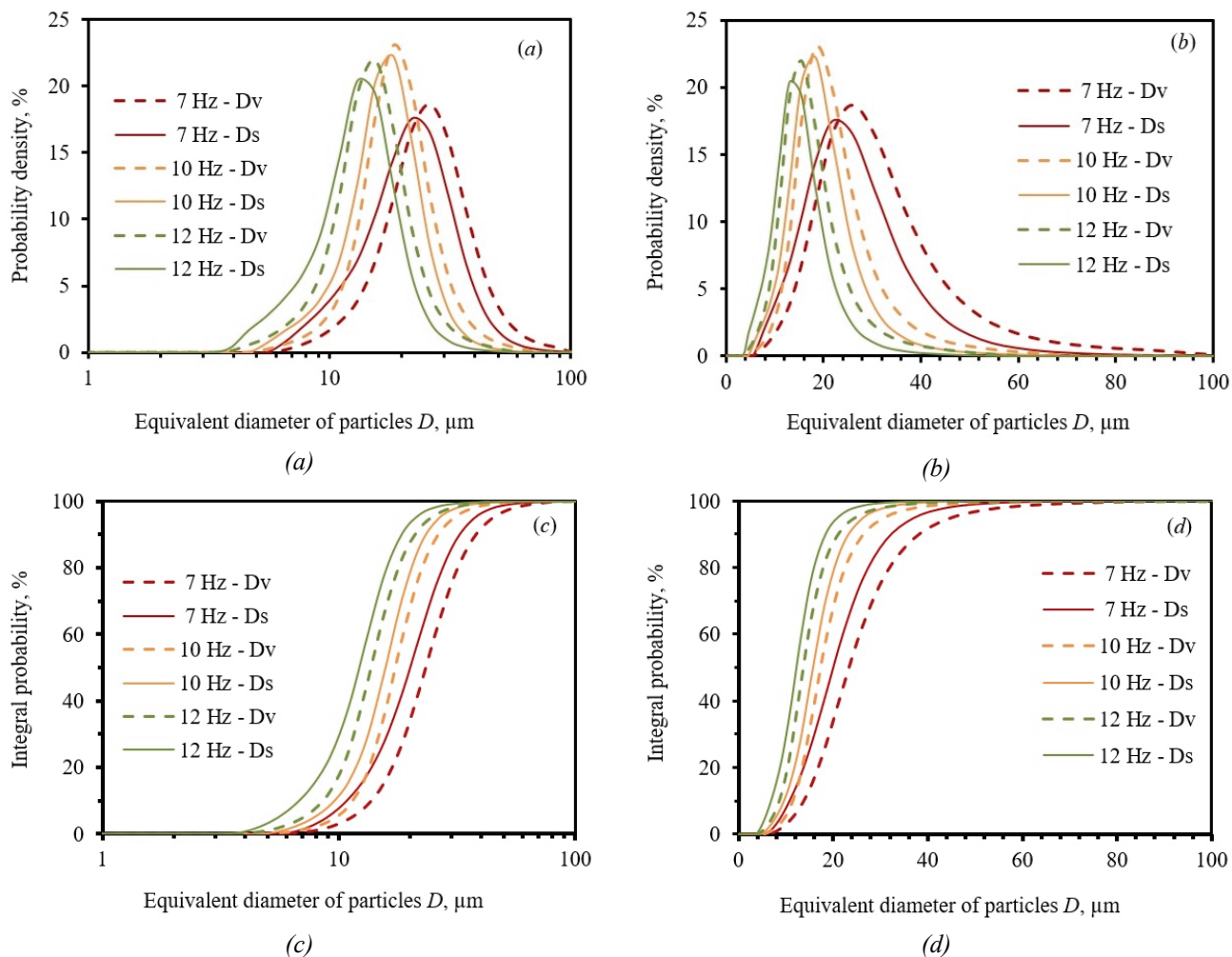


Fig. 1. The granulometric composition of the 29NK alloy powder produced at different air-centrifugal classification frequencies:

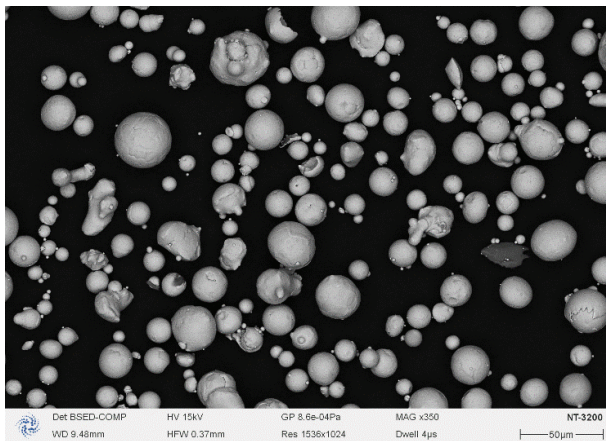
a – differential distribution; *b, d* – the same data, but presented in semi-logarithmic coordinates; *c* – integral distribution

Table 3. Main characteristics of the distribution of the granulometric composition of the 29NK alloy powder after air-centrifugal classification at different frequencies

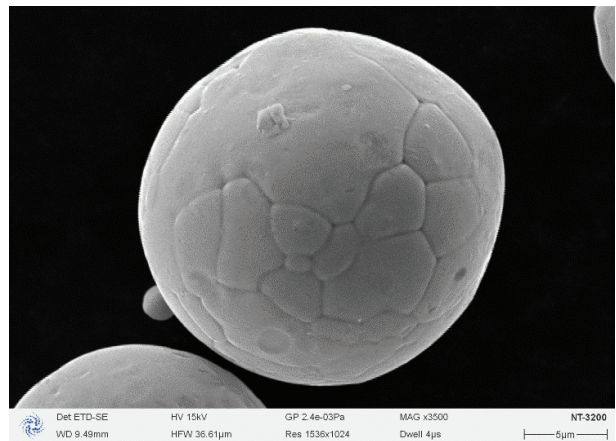
Characteristic	The value of the characteristic at different separation frequencies, Hz		
	7	10	12
Mode of distribution of diameters of spherical particles with equivalent surface area D_s , μm	21.19	16.33	12.63
Mode of distribution of diameters of spherical particles with equivalent volume D_v , μm	25.12	18.45	14.52
Standard deviation σ in the distribution of diameters of equivalent spherical particles, μm	9.60	5.92	4.93
Maximum diameter of spherical particles D_{90} , μm	88.0	62.22	52.32
Minimum diameter of spherical particles D_{10} , μm	6.54	5.50	3.89

In addition, Figs. 2*b*, 2*d*, 2*f* shows that the 29NK alloy powder has a clearly defined grain structure. With a decrease in the powder particle size, a decrease in the dendritic parameter should be observed, since due to the high surface area to volume ratio and the low mass of the fine (dust) fraction granules, it is this fraction that achieves the highest cooling rate and the most non-equilibrium structure, characterized by a very small dendritic parameter of the grains distributed over the surface of

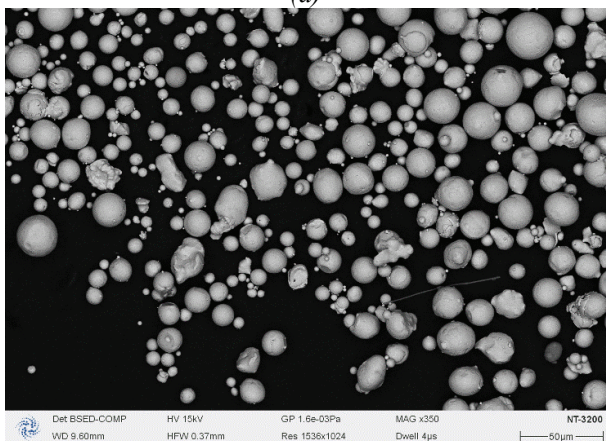
the granules. This feature of the microstructure, which promotes the localization of the distribution of the alloying components of the alloy precisely on the surface of the granule, is an additional favorable factor in the sintering of fine dust fractions. Sintering of fine powder fractions with a similar structure should contribute to the production of a monolithic material with a fine-grained structure, which provides an increase in the physical and mechanical characteristics according to Hall-Petch relation.



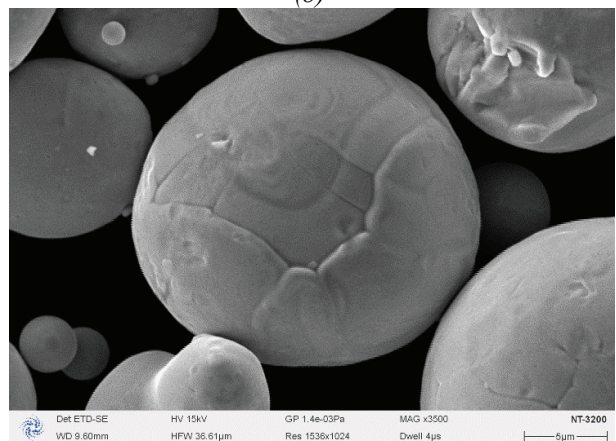
(a)



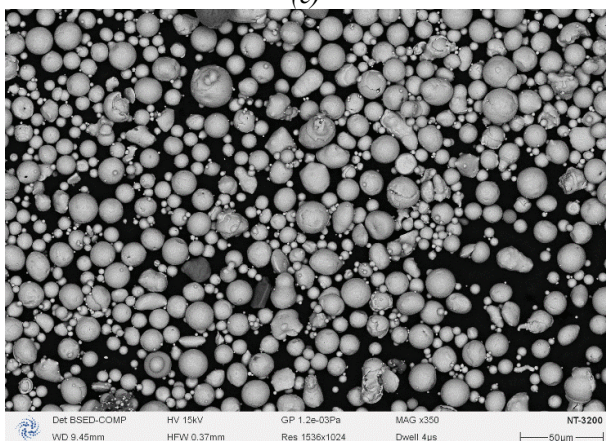
(b)



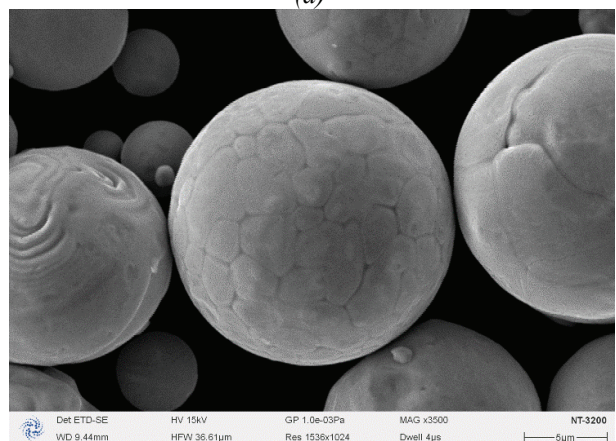
(c)



(d)



(e)



(f)

Fig. 2. Morphology of fine-grained particles of 29NK alloy powder obtained at different frequencies of air-centrifugal classification: *a*, *b* – at 7 Hz; *c*, *d* – at 10 Hz; *e*, *f* – at 12 Hz

Returning to the analysis of laser diffraction data on the granulometric composition of the powder fractions under study (Fig. 1), we can draw the following conclusion. For all three fractions, the differential distribution of particle sizes in Cartesian coordinates (Fig. 1a) is close to a lognormal distribution, which is confirmed by the normal distribution when switching to semilogarithmic coordinates (Fig. 1b). The lognormal nature of the distribution indicates that the process that generates this distribution has one dominant power law. The proven single-factor nature of the air-centrifugal classification process in relation to the conditions of this study allows us to construct single-factor mathematical models that approximately fit the experimental data phenomenologically.

As can be seen from the data presented in Table 1, with an increase in classification frequency, the particle size of the resulting fraction decreases. This is reflected in a decrease in both the mode of diameter distribution for spherical particles with equivalent surface area D_s and the mode of diameter distribution for spherical particles with equivalent volume D_v , as well as a decrease in maximum D_{90} and minimum D_{10} equivalent particle diameters.

The effect of air-centrifugal classification frequency on the granulometric composition characteristics of 29NK alloy powder fractions is illustrated in Fig. 3.

The linearity and almost identical tangent of the angle of inclination of the dependencies shown in semi-logarithmic coordinates in Fig. 3 indicate that they can be described by an approximative equation of the activation type. Moreover, these dependencies will have almost the same value of the effective activation energy.

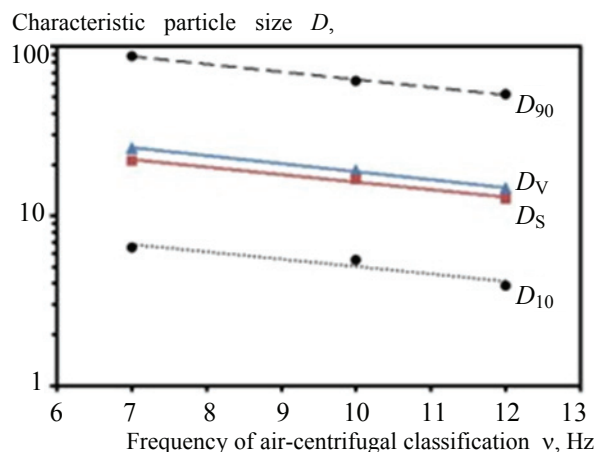


Fig. 3. The effect of the classification mode on the granulometric composition of the 29NK alloy powder fractions

This suggests that the classification models obtained take into account the influence of the particle size factor, but the power of this factor is extremely low. The analytical dependences obtained as a result of the approximation of the data shown in Fig. 3 are presented in Table 4.

In addition, it was found that with an increase in the classification frequency, not only a decrease in the size of the classified particles occurs, but also a decrease in the size spread of the classified powder (Fig. 4). The measure of the spread is the values of the standard deviation S and the RMS – root mean square deviation σ . This is due to the fact that despite the fact that the effective activation energy of the classification process is almost identical for particles of different sizes, this factor still affects the process, albeit with a small power.

Table 4. The main characteristics of the distributions of the granulometric composition of 29NK alloy powder after air-centrifugal classification at various frequencies

Characteristic	Approximating dependence	Determination coefficient R^2
Effect of the separation frequency ν on the value of the mode of diameter distribution of spherical particles with equivalent volume D_v and equivalent area D_s	$D_v(\nu) = 54.199 \exp(-0.109\nu)$	0.9982
	$D_s(\nu) = 43.9 \exp(-0.102\nu)$	0.9877
Effect of the separation frequency ν on the maximum D_{90} and minimum D_{10} diameter of equivalent spherical particles of 29NK alloy powder	$D_{90}(\nu) = 181.73 \exp(-0.105\nu)$	0.9944
	$D_{10}(\nu) = 13.684 \exp(-0.1\nu)$	0.9098
Effect of the separation frequency ν on the RMS value σ and the standard deviation S in the particle size distribution of 29NK alloy powder	$\sigma(\nu) = 0.1463\nu^2 - 3.7143\nu + 28.43$	1.0
	$S(\nu) = 0.1443\nu^2 - 3.6403\nu + 27.46$	1.0

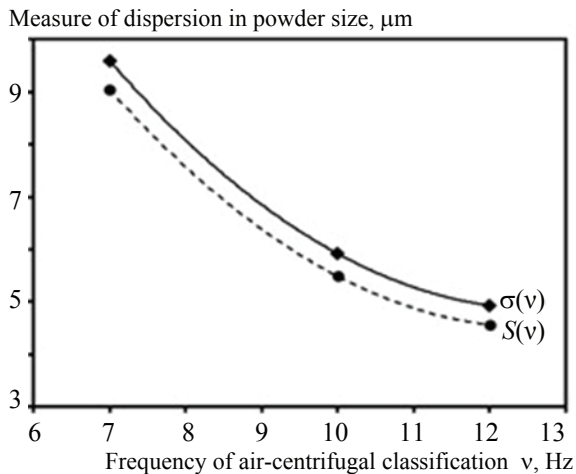


Fig. 4. Effect of classification frequency on the size distribution of 29NK alloy powder

It should be noted that the correctness of the obtained phenomenological models describing the effect of classification frequency on the main characteristics of the particle size distribution of the final powder fraction is based on the limitations used in this work. Thus, the same initial powder samples were used for classification; the duration of the classification process was also the same for all fractions studied and obviously ensured the completion of the process for the selected sample mass. Thus, the problem being solved excluded the time factor and was not considered as a kinetics problem. At the same time, the chosen formulation of the problem ensures the methodological possibility of constructing simple single-factor models of air-centrifugal classification in laboratory conditions for any pair of “powder with the initial particle size distribution – air-centrifugal classification device”. The disadvantage of the proposed approach is the impossibility of calculating the conversion of the particle size distribution of the classified powder at intermediate stages of the process until its complete completion.

4. Conclusion

The granulometric and morphological composition of 29NK alloy powder fractions obtained under different air-centrifugal classification conditions was studied using laser diffraction and SEM methods. The isoconversion model-independent analysis of granulometric composition was used to construct phenomenological dependences of the air-centrifugal classification frequency effect on the granulometric composition of the resulting powder fraction. It was shown that with an increase in the classification frequency, the particle sizes of the

resulting fraction decrease and the particle size distribution range decreases. The excess coefficient of the granulometric distribution also increases – it becomes “sharp”, approaching the single distribution of an ideal classifier. The microstructure of fine powder fractions should promote sintering of the material with a fine-grained structure, which ensures an increase in the physical and mechanical characteristics. Thus, the classification of dust fractions of powders can expand the use of powders with different fractional compositions, including fractions unsuitable for additive technologies.

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7. Conflict of interest

The authors declare no conflict of interest.

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