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Technology of obtaining composite conglomerate powders for plasma spraying of high-temperature protective coatings

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ABSTRACT

Introduction. For parts of gas turbines operating under conditions of corrosion-erosion and intense wear, the most acceptable are compositions containing Ni, Co, Cr, Al, B, Y both in pure form and in the composition of compounds applied on the contact surface during thermal spraying. Modern integrated complexes obtained by combining dissimilar substances in the form of a single composition are promising. Such powders are obtained either by cladding or by conglomeration of finely dispersed starting components into a larger particle. The problem of developing and manufacturing plants for conglomeration of powders is urgent and practically important, since it makes it possible to obtain material for thermal spraying of coatings for high-temperature purposes. **The aim of the work** is to develop a technological scheme for obtaining powders of the required chemical composition with a given particle shape and size, intended for spraying high-temperature protective coatings. **Materials and research methods**. A technology is developed for the production of integrated powders for spraying coatings using the method of spray drying and subsequent sintering in vacuum or in an argon-hydrogen atmosphere, which avoids the loss of feedstock due to the return of fine and coarse fractions. A technology for preparing materials for spray drying and granulation is proposed. A gravity type aerodynamic classifier is designed and manufactured, which makes it possible to select automatically the powder fraction necessary for spraying the coating, as well as return the unwanted fraction for recycling. The morphology of the granular powder is determined using a TESCAN scanning electron microscope. The chemical composition of the resulting integrated complexes is determined by X-ray microanalysis on an OXFORD attachment. **Results and discussion.** The technological conditions for obtaining powders of a given size (40…100 μm) are established. It is shown that the shape of the conglomerate particles after spray drying is close to spherical. On the basis of a multifactor experiment, the optimization of the technological process for obtaining powders Ni-17Cr-10Al-1Y and Ni-22Cr-16Al-1Y with sizes up to 100 μm is performed. It is shown that when conglomerating powders with increased aluminum content (Ni-22Cr-16Al-1Y), it is necessary to take into account the exothermic reaction of nickel aluminide formation and dilute the mixture of initial components before sintering with the finished sintered powder. The resulting integrated complexes are characterized by high heat resistance; therefore they are designed and successfully used for plasma spraying of protective coatings for high-temperature purposes. **Conclusions.** A technology is developed for obtaining composite conglomerated powders Ni-17C-10Al-1Y and Ni-22Cr-16Al-1Y with particle sizes up to 100 μm and a shape close to spherical. A distinctive feature of this technology is that it avoids the loss of feedstock by returning fine and coarse fractions.

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Introduction

In modern gas turbine construction, sprayed coatings of the Ni-Co-Cr-Al-B-Y system are considered the most effective for protecting parts operated under corrosion and erosion conditions and intensive wear [1–8]. The use of plasma spraying as a method of applying high-temperature protective coatings allows for local or additional hardening and protection of individual parts of the surface to give them special service properties. Certain technological difficulties arise when spraying dissimilar metal powders with significantly different melting temperatures and thermophysical properties. The resulting coatings are characterized by increased porosity, heterogeneous chemical composition, a tendency to crack formation, and poor adhesion to the surface of the part. The use of the so-called integrated complexes, which are a combination of dissimilar substances in the form of a single composition in each powder particle solves the problem of differences in thermophysical properties. The integrated complexes are created by either cladding one or several layers of other materials on the initial matrix particle, or by conglomerating finely dispersed initial components into a larger particle [11, 12]. The maximum particle size, their shape, and the stability of the granulometric and chemical composition are particularly important for obtaining integral compositions, so many researchers pay special attention to determining these characteristics. Systematic long-term studies have revealed the most acceptable compositions to be the ones that crystallize to form eutectic structures based on nickel and/or cobalt, and that combine high protective properties with satisfactory plasticity due to changes in the content and concentration of alloying elements [13,14]. Previous studies, including field tests, have shown that the best results were obtained in the case of using the developed integral composition of the composition Ni-22Cr-16Al-1uA [15-17].

The accumulated experience shows that for plasma spraying of high-temperature coatings, the particle size of integral complexes should correspond to the interval of 20 ... 80 μm. An important issue is the ability of powders not to create congestion in transport pipelines, their uniform supply to the plasma jet and free movement with the gas flow. This can be achieved with a spherical or similar particle shape [6, 11, 12]. The spray drying method used to intensify the drying and granulation processes of materials has proved effective [19-21]. In other countries, variations of this method are used to obtain powders for plasma spraying, for example, Al_2O_3-Mo, WC-Co [22-25].

The aim of the work is to develop a technological scheme for obtaining powders of the required chemical composition with a given particle shape and size intended for spraying coatings.

Research Methodology

The authors proposed a fundamentally new technological scheme for obtaining composite powders for spraying coatings, which includes several sequential operations (Fig. 1).

The developers designed and manufactured a plant for spraying suspensions, its scheme is shown in Fig. 2. The body of the plant's chamber *1* has the following dimensions: diameter 850 mm, height 3000 mm. Such dimensions of the chamber ensure complete drying of the powder and prevent it from sticking to the walls. This in turn enables obtaining fine powders in a shape close to spherical. The presence of the upper *2* and lower *3* inputs of the sprayer *4* in the chamber allows for the process of spraying suspensions to work in two directions: "top-down" and "bottom-up". According to the second option, the drying time of the drop in the air stream is significantly higher. However, there is significant turbulence of the air-drop flow, which increases the probability of liquid droplets' interaction with the walls of the chamber and increases the number of deformed and destroyed granules.

The flow rate of the suspension is set by the value of the air pressure in the feeder and the diameter of the nozzle outlet 1.3...1.2 mm. The flow rate of the spraying air is set similarly. The performance of the plant and the granulometric composition of the powder are largely determined by the spraying mode; with the set nozzle size, the mode depends on the pressure and temperature of the air that feeds and sprays the suspension.

The chamber has a door with a viewing glass *5* and a classifier *6*. It is installed on a stand *8* and is equipped with a feeder for suspensions *7*, a distribution panel *9*, and a spray air heater *10*.

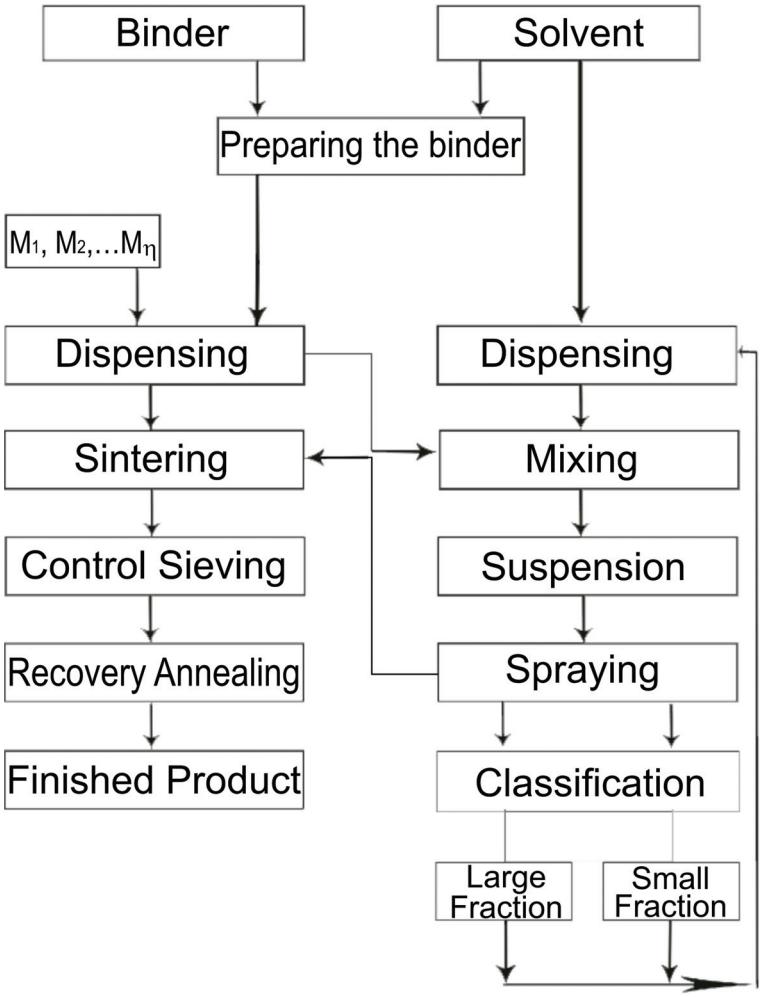

Fig. 1. Technological scheme for obtaining composite powders

Traditionally, a set of sieves with different cell sizes is used to determine the granulometric composition of the powder. An aerodynamic classifier of the gravity type was designed and manufactured to automate the process of sifting the powder, (Fig. 3). It is a system of axially arranged pipes. The air flow rate and the dimensions of the pipes are selected in such a way that there is a double classification of the powder into three fractions. Depending on which fraction needs to be used in the further powder preparation process, the remaining parts are sent to the head of the process, which ensures almost 100% powder use in the spray drying.

An important role in the spraying process is given to the choice of the spray nozzle type (Fig. 4). An analog of the nozzle we used was the one for dispersed fuel oil to the furnaces combustion chamber [22]. Such a nozzle allows obtaining the size of the sprayed fuel oil particles within 0 ... 100 μm. Since the viscosity of fuel oil and the suspensions we used is approximately the same, this nozzle design was applicable for our purposes. The total performance of the plant for conglomerating powders is 20 ... 50 kg of dried material per hour.

The first stage of the developed manufacturing process deals with preparing the suspension to be subsequently spray-dried. A solvent and binders for the suspensions are selected based on the properties of the resulting compositions, as well as granulation conditions and requirements for the purity of the resulting product. There are certain requirements for the solvent and binder: they should easily and, if possible, completely evaporate from the powder conglomerates under appropriate temperature conditions. The solvent evaporates from the suspension droplets during spraying and drying in the air stream. The binder is removed during the subsequent sintering of the composite particles. According to the authors [23], the binder should be stable at conglomeration temperatures and evaporate at approximately 50 K above these temperatures

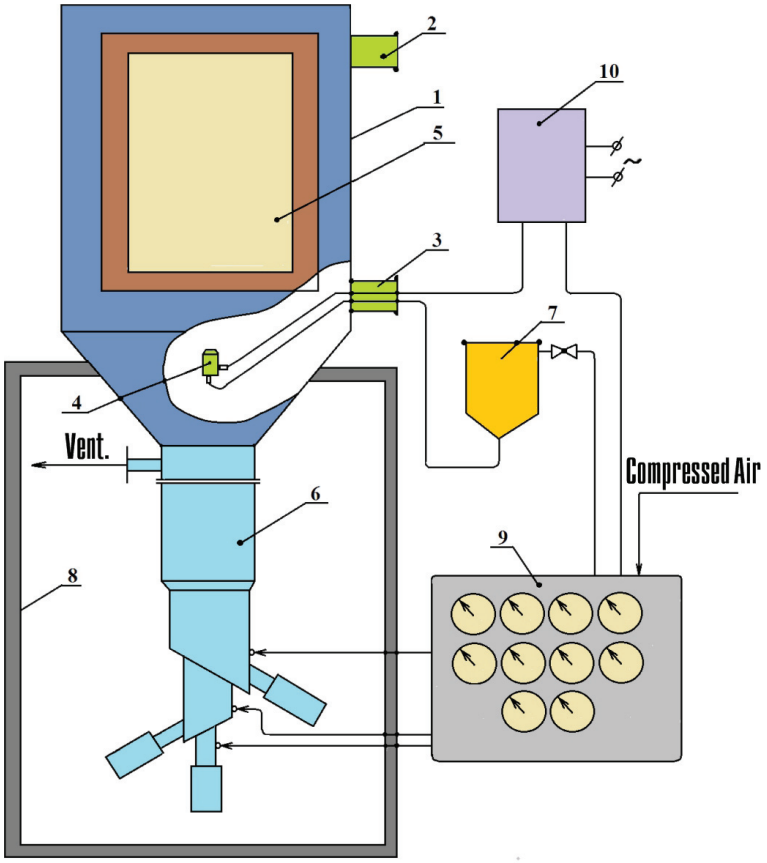

Fig. 2. Schematic diagram of a spray dryer for powder conglomeration

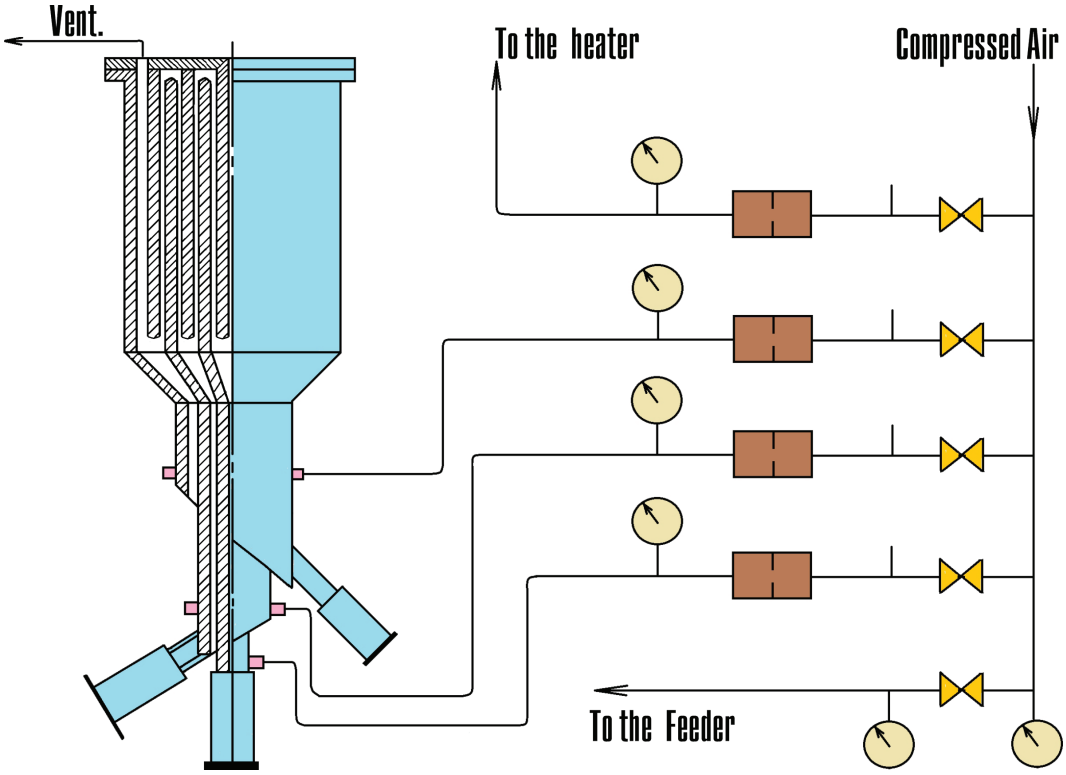

Fig. 3. Gravity Type Powder Classifier

and at least 50 K below the sintering temperature of subparticles in conglomerates. These requirements allow the use of a wide range of substances as a binder, for example, polyvinyl alcohol, stearic acid, paraffins, polyethylene glycol, and various resins.

In this work, for spray drying at room temperature, rubber and gasoline or polyvinyl acetate (PVA) and an equi-volume mixture of butyl acetate and acetone were used as a binder and solvent, respectively. The latter pair is preferred due to the higher evaporation of the solvent during the spraying process and the formation of strong conglomerates, which facilitates the classification of the obtained powders. The indisputable advantage of using PVA, unlike rubber, is the possibility of its almost complete removal during the sintering of subparticles in conglomerates.

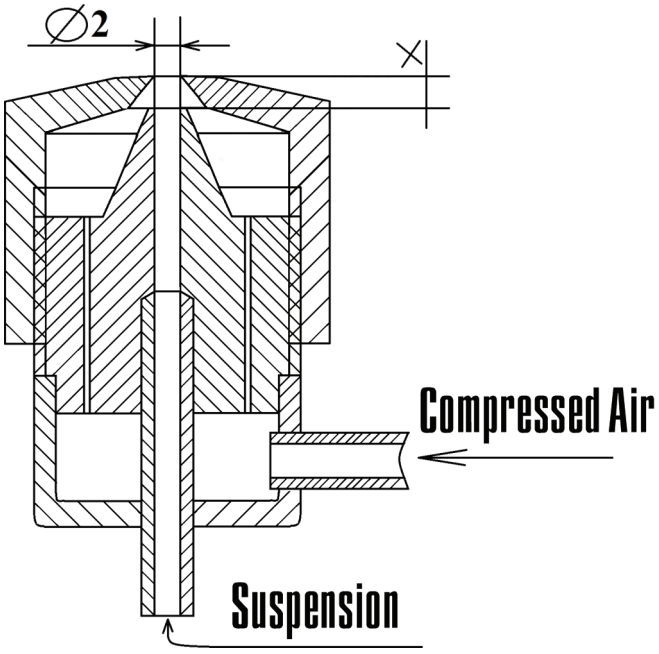

Fig. 4. Suspension spray nozzle

The amount of binder introduced into the suspension significantly affects its stability before being fed to the spray nozzle, as well as the strength of the resulting granules of composite materials. In each specific case, we experimentally determined the amount of the binder introduced into the charge from the initial fine powders, its value varying within 0.5...1.5% of the powder weight. The amount of solvent should be minimal for the mobility of the suspension to be preserved; it is also determined experimentally. The granulometric composition of the powders ultimately depends on the content of the binder and solvent in the suspension, as well as the type of spraying device and the conditions of spraying. The initial suspensions used for spraying have a solid component concentration of 70...85% depending on the density and dispersion of the integrated complexes. The introduction of additives (surfactants or electrolytes) is recommended in case of insufficient stability and mobility of the suspension [24].

To obtain conglomerates corresponding to the given chemical composition, the suspension was prepared by thoroughly mixing the initial charge with a solvent and a binder in mixers of various designs for a sufficient time, varying depending on the composition and the characteristics of the starting materials from 2 to 8 hours. The suspensions with small amounts of alloying additives (up to 2%), e.g. tantalum, yttrium, and niobium were mixed for a longer time.

The morphology of the integrated complexes was determined using a TESCAN scanning electron microscope, whose software capabilities allow to automatically determine the particle size. The chemical composition was determined by the X-ray microanalysis of powder particles using an OXFORD energy dispersion spectrometer. Averaging was performed on 20 particles in each of the five samples. The resistance of the obtained integrated complexes to high-temperature oxidation was determined by the changes in the specific mass (ΔM) of the powder sample after exposure in an alundum crucible in an electric resistance furnace at a temperature of 1324 K for 25 hours. The fluidity of the conglomerated powders was determined according to the requirements of the state standard GOST 20899–98.

Results and discussion

The dispersion of the initial materials is important for conglomerating powders using the spray drying method. The size of the subparticles of these powders should not exceed 1/5 of the conglomerates' diameter [25]. Reducing the size of the subparticles, firstly, increases the mobility and stability of suspensions. Secondly, since very small starting materials are used, a more uniform distribution of all the constituent components in the finished powder is achieved, especially during micro-alloying. Thirdly, conglomerates with a close to spherical shape are formed from small subparticles which are less susceptible to mechanical

destruction during classification after sputtering and which have greater fluidity. Fourth, the sintering surface of the subparticles in conglomerates increases, and as a result, their strength increases as well. With optimal sintering of the subparticles, the maximum density of conglomerated particles is achieved. By adjusting the size of the subparticles, the density of the powder can be controlled within the accepted limits.

The formation of composite particles (conglomerates) occurs in the process of suspensions spray drying. The size of the forming particles depends on the subsequent sintering. The granulometric composition, fluidity of the resulting powder, and its mechanical strength depend on the solvent and binder used, their concentration, the size of the initial powders, the airflow rate and the sprayed suspension, the temperature of the spraying air, and the type of nozzle used.

According to [26], the average diameter of the sprayed particles can be determined from the empirical equation

$$d = 585\frac{\sqrt{\sigma}}{U\gamma_p} + 507\left(\frac{\eta}{\sqrt{\sigma\gamma_p}}\right)^{0.45}\left(\frac{1000G}{V}\right)^{1.5}\mu m, \quad (1)$$

where U is the relative velocity between the solution and the gas, m/s; G and V are the flow rate of the solution and gas, m^3/s, respectively; η is the viscosity coefficient of the solution, P; σ is the surface tension of the solution, dyn/cm; γ_p is the density of the solution, g / cm^3.

Equation (1) demonstrates that obtaining smaller powders particles necessary for plasma spraying in a dynamic vacuum requires increasing the speed of the spraying air and the density of the suspension used, as well as reducing the surface tension and viscosity of the solution. The decrease in the viscosity of the suspension, in turn, is achieved by reducing the concentration of the binder and increasing the amount of the solvent.

To isolate the necessary fraction of the powder, the latter was classified in sieves. The waste of small and large fractions is returned to the process at the stage of the suspension preparation. The fractionated powders were sintered in a free backfill in vacuum or argon under the following conditions: the binder (PVA) was distilled at 573...723 K, after which the temperature was increased to such values when the subparticles sintered inside the conglomerates, while the conglomerates did not sinter with each other. The temperature and sintering time for each powder composition were determined experimentally.

The results of the research have shown that, on average, the sintering temperature of conglomerated powders should be set at 100 ... 250 K below the sintering temperature of compact materials. The optimal conditions for sintering conglomerated powders are achieved when the sintered mass of the powder is a briquette that easily collapses when crushed. After the control sieving, which is necessary to remove fine particles formed during the destruction of individual granules and large sintered conglomerates, the powder is ready for spraying. The shape of the integrated powders particles after spray drying conglomeration is close to spherical (Fig. 5). According to the data of X-ray microanalysis, the composition of the obtained integral complexes was as follows: 1) 71 wt. % Ni, 17 wt. % Cr, 10 wt. % Al, 1 wt. % Y and 2) 61 wt. % Ni, 22 wt. % Cr, 16 wt. % Al, 1 wt. % Y.

If large batches of powders are conglomerated according to the full technological scheme (see Fig. 1) with the return of small and large fractions, the yield of fractions allocated for spraying can be close to 100 %. Thus, obtaining clean-cut fraction powder materials for gas-thermal spraying becomes highly efficient.

The spray drying process was optimized to obtain a conglomerate powder of the composition Ni-22Cr-16Al-1Y with a grain size of 0 ... 100 μm. A complete 2^3 factorial experiment was used. Factors: the amount of solvent per 1 kg of mixed powder V ml/kg; excess pressure in the feeder with the suspension P, atm. ; the height of the nozzle cut above the place where the suspension is injected into the gas stream, X, mm. The spray air consumption was 0.5^{m^3}/min. Insufficient airflow leads to the powder sticking to the walls of the chamber. The powder that falls off the chamber walls as it dries has poor sphericity and reduces the quality of the final product. The air pressure inside the pipes of the aerodynamic classifier was selected in such a way that the powder with a grain size of more than 100 μm was deposited in the central pipe, and the

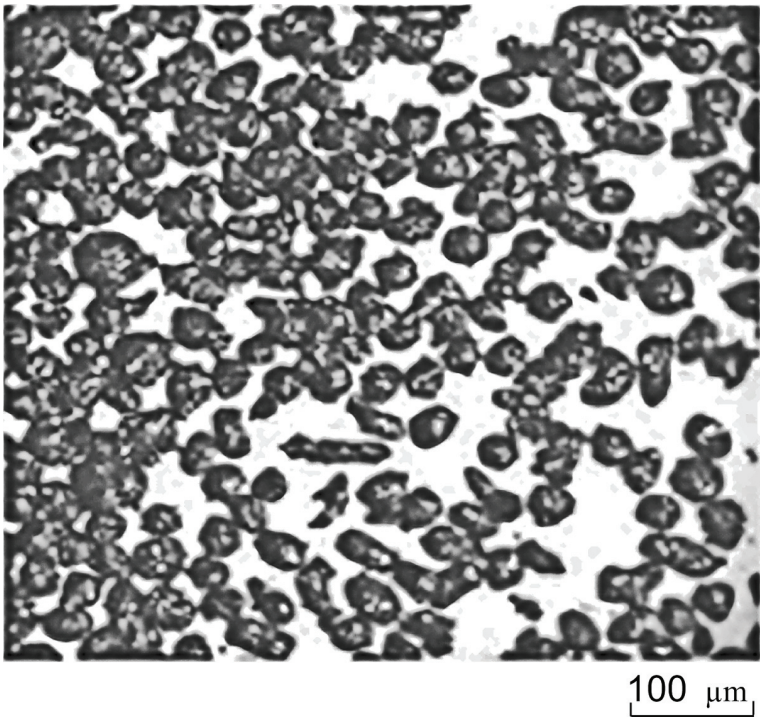

Fig. 5. Appearance of conglomerated powders for spraying Ni-22Cr-16Al-1Y composition

powder with grain size 0...100 μm was deposited in the two side pipes. The coarse powder was sent to the head of the process. The fraction content of 0...100 μm in the "dry" powder was taken as the optimization parameter (*Y*). Table 1 shows the values of the factors in absolute and encoded form, and the results of the study are also presented here.

Taking into account the significance of the coefficients, the regression equation has the following form (the factors are presented in the encoded form):

$$Y = 66.4 + 11.6X + 10.2VX, \%. \quad (2)$$

The analysis of the regression equation shows that obtaining the maximum amount of powder with a grain size of 0...40 μm requires increasing *X* and *V*. The pressure in the suspension feeder within the studied interval practically does not affect the composition of the dispersed powder. The increase in the yield of the fraction 0...100 μm, depending on *X* and *V*, is explained by the fact that the increased solvent content in the suspension reduces its viscosity and surface tension, thus contributing to the jet fragmentation into smaller particles at spraying. When the solvent and the sprayed suspension are used excessively, the powder adheres to the walls of the spray chamber. A high value of the size *X* leads to additional jet fragmentation inside the nozzle, resulting in an increased number of smaller particles. According to Table 1, the maximum powder yield of 0...100 μm is obtained under the following conditions: *V* = 380 ml of solvent /1 kg of powder; *X* = 2.0 mm; *P* = 1.32 atm.

The study of the spraying air temperature effect on the quality of the sprayed powder showed that at temperatures of 363...523 K, the stability of the spraying process sharply decreases, and the process is interrupted in 1...2 minutes after the start due to the drying of the suspension in the nozzle feed channel.

The binder (PVA) was distilled at a temperature of 573...773 K for half an hour, then the temperature was increased to the values of the subparticles sintering inside the conglomerates. An exothermic reaction of Al and Ni interaction with a large release of heat begins at the temperature of 903...923 K. The temperature of the crucible with the powder briefly rose to 1023...1273 K. To prevent sintering of conglomerates among themselves, they were diluted with a sintered powder of the same composition in an amount of 25 ... 35 %. After the solid-phase interaction, isothermal annealing was carried out at 1073 K for 30 min.

Table 1

Influence of spray drying and subsequent sintering modes on the particle size distribution of the resulting powders

Experiment number	Spray mode						The fraction 0 ... 100 μm yield, %	Powder yield after sintering (fraction 0..100 μm), %				Durability of powders (10 ... 40 μm) Ni-22Cr-16Al-1Y against oxidation in air at 1323 K (weight gain for 6 h), mg/g
	V, ml/kg		*X*, mm		*P*, atm							
	abs	enc	abs	enc	abs	enc		0..40 μm	40..63 μm	63..100 μm	>100 μm	
1	380	+	2	+	1.32	+	92.2	12.8	15.8	48.3	23.1	107.9
2	360	–	2	+	1.32	+	62.2	21.6	9.4	51.8	17.2	39.8
3	380	+	1	–	1.32	+	50.0	8.4	17.2	51.9	22.5	77.4
4	360	–	1	–	1.32	+	68.6	23.5	15.6	45.6	15.3	91.8
5	380	+	2	+	1.08	–	80.2	20.2	20.5	46.6	12.7	48.0
6	360	–	2	+	1.08	–	79.4	23.8	16.6	39.9	19.7	108.0
7	380	+	1	–	1.08	–	35.4	7.9	19.4	55.8	16.9	45.3
8	360	–	1	–	1.08	–	68.0	20.6	15.6	47.0	1.08	68.0

Table 2 presents the granulometric composition of the sintered powders. The bulk of the resulting powder has a size greater than 40 μm. The yield of the 0...40 μm fraction is less than 25 %. Obtaining the powder used for spraying in a dynamic vacuum requires sintering a smaller fraction of 0...40 μm. The obtained results demonstrate that sintering a powder with a grain size of 0 ... 40 μm leads to a significant powder grain size increase due to partial sintering of conglomerates among themselves, which deteriorates the morphology of the final product. This is due to a larger contact area than in the case of 40 ... 100 μm fraction sintering.

Table 2

Influence of the initial particle size distribution and chemical composition on the powder after sintering

Chemical composition of powders, masses.%	Initial size of powders, μm	Powder yield after spraying, %	Granulometric composition after sintering, %			
			0..40, μm	40..63, μm	63..100, μm	>100 μm
Ni-17Cr-10Al-1Y	0…100	–	29.2	36.0	28.1	5.6
Ni-22Cr-16Al-1Y	0…100	–	24.0	37.6	27.4	11.0
	0…40	35.8	61.4	32.3	6.6	–
	40…100	64.2	7.7	35.1	44.1	1.3

The yield of fractions after sintering is also affected by the composition of the powder. Thus, sintering a powder of the Ni-17Cr-10Al-1Y composition yields a smaller amount of a large fraction than sintering a powder of the Ni-22Cr-16Al-1Y composition, which is associated with a lower Al content and, as a result, less heat released during the sintering reaction and a lower sintering temperature (Table 2).

After sintering the powder, it is divided into fractions and recovery-annealed. The sieving of 40 ... 100 μm fraction is carried out using a standard set of sieves, and the separation of the fraction with a size of 10 ... 40 μm was carried out by washing the powder three times in distilled water, followed by drying for 2...3 hours at a temperature of 373...383 K. Finishing treatment of the powder is recovery annealing in a hydrogen atmosphere at 873 K for half an hour to remove oxides present in the initial powders and formed during the preparation of conglomerates.

To assess the quality of the obtained powders, their resistance to oxidation in the air was measured at 1323 K. The test results can be described by a regression equation:

$$\Delta M = 73,3 - 3,63V + 2,65X + 5,95P + 5,65VX + 17,05VP - 8,03XP + 14,95XVP, \quad (3)$$

According to the experimental data, to improve the quality of the powder, the amount of solvent (V) in the sprayed suspension should be reduced; X and the excess pressure in the chamber (P) should be increased. In practice, as a rule, the volume of the solvent tends to be reduced. Thus, to obtain powders for plasma spraying in a dynamic vacuum, we determined the following technological regulations: V = 360 ml/1 kg of powder; X = 2.0 mm; P = 1.28 atm.

Powders obtained with the sprayed air heating are characterized by lower heat resistance, for example, for 363 K, it is 170.9 mg/g, and for 523 K, it is 208.6 mg/g. This indicates that the accelerated drying of conglomerates occurs during the spraying process, leading to deformation and increased porosity of the particles.

We conducted a comparative properties study of the powders obtained using spray drying and spraying of melt in a vacuum (Table 3).

Both methods, applied to plasma spraying, give powders that are similar in properties. However, the spray drying method is cheaper and more universal in terms of the powders produced. The chemical composition of the powder obtained by the spray drying method practically does not differ from the composition of the initial components.

Table 3

Comparative characteristics of powders obtained by spray drying and spraying of melts

Powder Property	Method of obtaining	Size of powders, μm					
		0…40	40...50	50...63	63...100	100...200	>200
Content, mass.%	Spray drying	61.4	16.3	16.0	6.6	0	0
	Spraying of melts	50.5	24.2	13.4	9.1	1.8	0.9
Fluidity, sec	Spray drying	69.9	34.2	28.8	23.5	–	–
	Spraying of melts	57.8	25.3	28.0	31.5	–	–

To give the sprayed conglomerates sufficient strength and density, they are sintered, which includes the stages of removing the organic binder and sintering the subparticles in the conglomerate. The process of sintering the sprayed powder has a significant impact on the properties of the resulting product. The morphology, strength of the particles, and their granulometric composition largely depend on the conditions of the process.

After spray drying, the powder is placed in a free filling in a vacuum oven. Next, the furnace is degassed and filled with argon to the pressure of 0.1 MPa. Then the temperature is gradually raised at a speed not higher than 3...4 K $\times min^{-1}$ to remove the binder. If polyvinyl acetate (PVA) is used as a binder, the temperature of its complete removal from the powder mass is approximately 820 K. Then the working space of the furnace is degassed to a residual pressure of 0.1 Pa and heated to sintering temperatures. The sintering process can also be carried out at atmospheric pressure, for example, in an argon-hydrogen gas medium. In this case, the degree of powder oxidation is significantly reduced.

There are certain differences in the sintering process of Ni-Cr-Al-Me powders, where Me is the alloying additives of one or several rare elements, such as Y, Ta, Nb. These conglomerate powders are exothermic due to the reaction of nickel aluminides formation. The synthesis reaction begins when the powders are heated to 900...950 K, with the temperature in the reaction space of the furnace rising to 1500 K. In these conditions, conglomerates are strongly sintered among themselves, and this is unacceptable according to the technology. To reduce the sintering temperature of the powders, the sprayed powder was diluted in a certain ratio with a ready-made sintered powder of the same composition before placing it in the furnace. The degree of dilution ultimately determines the heating temperature of the powder and, as a result, the density and mechanical strength of the sintered conglomerates. To obtain strong and dense powder granules of the Ni-Cr-Al-Me composition with different content of chromium (20...28 %) and aluminum (12...20%), the optimal amount of diluent powder was determined to be within 20 to 40 %. For example, a powder of the composition (mass. %): Ni-28Cr-20Al-Me is best sintered with a dilution of up to 40 %.

After the exothermic reaction, the temperature in the furnace is set to 1020 K and maintained for half an hour until the subparticles are completely sintered in conglomerates. The result of sintering is a briquette that is easily crushed into individual granules. Control sieving is performed to separate small powder particles formed as a result of the individual granules' destruction and large conglomerates sintered together. The powder fraction suitable for spraying is subjected to recovery annealing in a hydrogen atmosphere at the

temperature of 800 K. Importantly, small amounts of alloying elements practically do not affect the powder sintering modes.

Finally, the resulting composite conglomerated granular powder material was used to create a combined heat-resistant coating to protect high-temperature parts of a gas turbine propulsion system. The coating consists of three layers. The first 28Al-2Si-1P3M layer with a thickness of 40 ... 55 μm is applied by the thermal diffusion method and located at the boundary with the substrate alloy [15]. This layer provides high heat and corrosion resistance, as well as diffusion stability of the entire coating. The second layer is the plasma sprayed one of Ni-22Cr-16Al-1Y composition with a thickness of 115 μm providing high-temperature resistance of the coating, as well as good surface adhesion of the ceramic outer layer. The third one is the heat-shielding outer ceramic layer of ZrO_2+Y_2O_3 composition with a thickness of about 50 μm; it reduces the surface temperature of the alloy during operation.

Conclusion

A technology for producing composite conglomerate powders for plasma spraying of high-temperature protective coatings has been developed. A distinctive feature of this technology is the method of spray drying and subsequent sintering in a vacuum or an argon-hydrogen gas medium.

To determine the granulometric composition, an aerodynamic classifier of the gravity type was designed and manufactured; the classifier automatically sorts the powder into fractions.

The proposed technology allows for obtaining powders of the given chemical composition with a particle shape close to spherical and dimensions within 40 ... 100 μm. Integrated powder complexes of the following chemical compositions were obtained: 1) 71 wt. % Ni, 17 wt. % Cr, 10 wt. % Al, 1 wt. % Y; 2) 61 wt. % Ni, 22 wt. % Sg, 16 wt. % Al, 1 wt. % Y.

The research demonstrates that sintering powders of the Ni-Cr-Al-Y composition should make provisions for the exothermic reaction of nickel aluminides formation, which begins when the powders are heated to 900...950 K. Obtaining an integrated powder complex with a high content of aluminum (Ni-22Cr-16Al-1Y) is proposed to perform by dilution with a ready-made sintered powder of the same composition before loading into the furnace.

References

1. Rickerby D.S., Winstone M.R. Coatings for gas tyrbines. *Journal of Manufacturing Processes*, 1992, vol. 7, iss. 4, pp. 485–526. DOI: 10.1080/10426919208947439.

2. Burgel R. Coating service experience with industrial gas turbines. *Journal of Materials Science and Technology*, 1986, vol. 2, pp. 302–308. DOI: 10.1179/mst.1986.2.3.302.

3. Guzanov B.N., Kositsyn S.V., Pugacheva N.B. *Uprochnyayushchie zashchitnye pokrytiya v mashinostroenii* [Reinforcing protective coatings in engineering industry]. Ekaterinburg, Ural Branch of the RAS Publ., 2003. 244 p. ISBN 5-7691-1405-3.

4. Chaki T.K., Singht A.K., Sadananda K. Effect of Co-Cr-Al-Y coating on microstructural stability and creep behavior of nickel-base superalloy. *Thin Solid Films*, 1989, vol. 168, pp. 207–220. DOI: 10.1016 / 0040-6090 (89) 90007-2.

5. Muboyadzhan S.A., Lesnikov V.P., Kuznetsov V.P. *Kompleksnye zashchitnye pokrytiya turbinnykh lopatok aviatsionnykh GTD* [Complex protective coatings for turbine blades of aircraft gas turbine engines]. Ekaterinburg, Kvist Publ., 2008. 208 p. ISBN 5-900474-60-7.

6. Kulik A.Ya., Borisov Yu.S., Mnukhin A.S., Nikitin M.D. *Gazotermicheskoe napylenie kompozitsionnykh poroshkov* [Gas thermal spraying of composite powders]. Leningrad, Mashinostroenie Publ., 1985. 199 p.

7. Uusitalo M.A., Vuristo P.M.J., Mantyla T.A. Elevated temperature erosion-corrosion of thermal sprayed coatings in chlorine containing environments. *Wear*, 2002, vol. 252, iss. 7–8, pp. 586–594. DOI: 10.1016/S0043-1648(02)00014-5.

8. Lai G.Y. *High-temperature corrosion and materials application*. Materials Park, Ohio, ASM International, 2007. 461 p. ISBN 978-0-87170-853-3.

9. Sidhu S.S., Prakash S. Performance of NiCrAlY, Ni-Cr, Stellite-6 and Ni_3Al coatings in Na_2SO_4-60% V_2O_5 environment at 900° under cyclic conditions. *Surface and Coatings Technology*, 2006, vol. 201, iss. 3–4, pp. 1643–1654. DOI: 10.1016/j.surfcoat.2006.02.035.

10. Matthews S., Schweizer M. Optimization of arc-sprayed Ni-Cr-Ti coatings for high temperature corrosion applications. *Journal of Thermal Spray Technology*, 2013, vol. 22, iss. 4, pp. 538–550. DOI: 10.1007/s11666-013-9914-y.

11. Kalinovskii V.R., Sokolov Yu.V., Il'yushchenko A.F., Troshchii V.V., Kalinovskii A.V. Konglomerirovannye kompozitsionnye poroshki dlya gazotermicheskogo napyleniya [Conglomerated composite powders for gas-thermal spraying]. *Perspektivy razvitiya poverkhnostnogo i ob"emnogo uprochneniya splavov* [Prospects for the development of surface and volumetric hardening of alloys]. Ed. by L.G. Voroshin. Minsk, 2004, pp. 124–133.

12. Ilinkova T.A., Barsukova E.A., Tagirov A.T. Vzaimosvyaz' kharakteristik poroshkovykh materialov i mekhanicheskikh svoistv plazmennykh teplozashchitnykh pokrytii [The relationship between the characteristics of powder materials and the mechanical properties of plasma heat-shielding coatings]. *Vestnik tekhnologicheskogo universiteta = Bulletin of the Technological University*, 2015, vol. 18, no. 15, pp. 116–121.

13. Kositsyn S.V., Guzanov B.N., Aleksin S.D., Kopylov A.A. Osobennosti struktury i svoistv γ+β splavov Ni-Cr-Al vblizi evtekticheskogo sostava [Features of the structure and properties of γ + β alloys Ni-Cr-Al near the eutectic composition]. *Fizika metallov i metallovedenie = The Physics of Metals and Metallography*, 1990, no. 9, pp. 114–122. (In Russian).

14. Tingaud D., Nardou F. Influence of non-reactive particles on the microstructure of NiAl and NiAl–ZrO_2 processed by thermal explosion. *Intermetallics*, 2008, vol. 16, iss. 5, pp. 732–737. DOI: 10.1016/j.intermet.2008.02.016.

15. Pugacheva N.B. Current trends in the development of heat-resistant coatings based on aluminides of iron, nickel and cobalt. *Diagnostics, Resource and Mechanics of Materials and Structures*, 2015, iss. 3, pp. 51–82. DOI: 10.17804/2410-9908.2015.3.051-082. Available at: https://www.dream-journal.org/DREAM_Issue_3_2015_Pugacheva_N._B._051_082..pdf (accessed 14.01.2021).

16. Pugacheva N.B., Guzanov B.N., Obabkov N.V., Bykova T.M., Michurov N.S. Studying the structure and adhesion strength of thermal barriers coating. *AIP Conference Proceedings*, 2019, vol. 2176, pp. 030013-1–030013-4. DOI: 10.1063 / 1.5135137.

17. Guzanov B.N., Pugacheva N.B., Alekseev V.D., Slukin E.Yu. Osobennosti sozdaniya kombinirovannykh teplostoikikh pokrytii dlya detalei vysokotemperaturnogo naznacheniya [Features of the creation of combined heat-resistant coatings for high-temperature parts]. *Vestnik Permskogo natsional'nogo issledovatel'skogo politekhnicheskogo universiteta. Mashinostroenie, materialovedenie = Bulletin PNRPU. Mechanical engineering, materials science*, 2020, vol. 22, iss. 3, pp. 12–19. DOI: 10.15593 / 2224-9877 / 2020.3.02.

18. Belopol'skii M.S. *Sushka keramicheskikh suspenzii v raspylitel'nykh sushilkakh* [Drying of ceramic suspensions in spray dryers]. Moscow, Stroiizdat Publ., 1972. 126 p.

19. Polyakov A.A., Kruglitskii N.N. *Raspylitel'naya sushka v tekhnologii radioelektronnykh materialov* [Spray drying in radio electronic materials technology]. Moscow, Radio i svyaz' Publ., 1982. 70 p.

20. Lykov M.M., Leonchik B.I. *Raspylitel'nye sushilki* [Spray dryers]. Moscow, Mashinostroenie Publ., 1966. 331 p.

21. Port D.J., Lafferty W.D., Cheney R.F. *Cermet plasma flame spray powder, method for producing same and articles produced the reform*. Patent USA, no. 3960545, 1976.

22. Cheney R.F., Lafferty W.D., Long G. *Tungsten carbide-cobalt flame spray powder and method*. Patent USA, no. 4025334, 1977.

23. Cheney R.F., Lafferty W.D., Long G. Free flowing, sintered refractory agglomerates. Patent USA, no. 3881911, 1975.

24. Stecura S. Effects of yttrium, aluminum and chromium concentrations in bond coatings on the performance of zirconia-yttrium thermal barriers. *Thin Solid Films*, 1980, vol. 73, iss. 2, pp. 481–489. DOI: 10.1016 / 0040-6090 (80) 90521-0.

25. Mrdak M.R. Mechanical properties and metallographic analysis of plasma spray ABS – Ni5.5wt.%Al5wt.%Mo coatings. *Vojnotehnički glasnik / Militaru technical courier*, 2019, vol. 67, iss. 3, pp. 573–587. DOI: 10.5937/vojtehg67-17424.

26. Vitman L.A., Kandelson B.D., Paleev V.I. *Raspylivanie zhidkosti forsunkami* [Spraying liquid with nozzles]. Moscow, Gosenergoizdat Publ., 1962. 264 p.

Conflicts of Interest

The authors declare no conflict of interest.