Physicochemical properties of lead brass charge obtained by electroerosion in isopropyl alcohol under optimal dispersion regimes

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In the work scientific and applicative principles of LS58-3 ($CuZn_{39}Pb_3$) grade brass waste processing technology into powders suitable for industrial application are realized. The purpose of the work was to study the physicochemical properties of the lead brass charge obtained by electroerosion in isopropyl alcohol under the optimal dispersion regimes. Grinding of alloy waste to microparticles was carried out on a special patented unit. Optimization of lead brass electrodispersing process was carried out by setting up a full factor experiment. The validation of composition and properties of the obtained powders was carried out using modern intercomplementary methods of physical materials science. It has been experimentally established that the composition, structure and properties of the charge dispersed by electrical erosion of brass LS58-3 are influenced by the chemical composition of the dielectric fluid, as well as dispersion modes. The particles of the resulting charge have a given complex of properties and can be used by various methods of powder metallurgy. The results of the conducted researches allow to recommend the use of the obtained powder as an initial raw material for obtaining alloy blanks and to expand the area of their practical application. Economic efficiency from the use of electroerosion powders is due to the use of waste and low energy-intensive technology for their production.

Key words: brass waste, electroerosion, powder, powder metallurgy.

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Introduction

Powder metallurgy is a branch of metallurgy, with the help of which it is possible to manufacture products of various shapes, purposes, as well as to create new materials with properties that cannot be obtained by traditional methods of processing [1–4].

The industry is currently facing problems of metal scarcity, the list of which includes copper and many alloying elements of alloys based on it. In particular, lead-added brasses improve machinability and antifriction properties, so they are widely used for the manufacture of friction parts. The addition of zinc increases corrosion resistance. However, due to the scarcity of lead and zinc, their content should be reduced and, if possible, they should be replaced by cheaper elements [5–8]. The solution to this problem may be the recycling of brass containing scarce components [14–19].

The problems of domestic recycling are related to the fact that so far these processes affect only a small part of technogenic resources. As studies [9–13] have shown, it is necessary to orient the prospective development of the Russian metallurgy to a wider use of secondary resources and a more active implementation of recycling processes, to improve the efficiency of raw material utilization. Implementation of recycling processes is one of the main factors of preservation and effective utilization of mineral and raw material potential both on the scale of a single country and on the global scale.

A promising but little-studied method of recycling any conductive materials is the method based on electrical erosion – electrical erosion dispersion. With its help it is possible to process metals to micro- and nanofractions particles with a given complex of physical and chemical properties with the possibility of their reuse [14–22]. The electric discharge method of processing is characterized by zero waste, energy efficiency and environmental friendliness of the dispersing process. However, this method is rarely used in industry due to the poorly studied composition, structure and properties of the resulting raw materials.

Therefore, the processing of brass waste by electroerosion and the study of composition, structure and properties of the charge on its basis is relevant.

The aim of the present work was to study the physicochemical properties of lead brass charge obtained by electroerosion in isopropyl alcohol under optimal dispersion regimes.

Materials and methods of research

Grinding of alloy waste to microparticles was carried out on a special patented unit [21] **(Fig. 1)**.

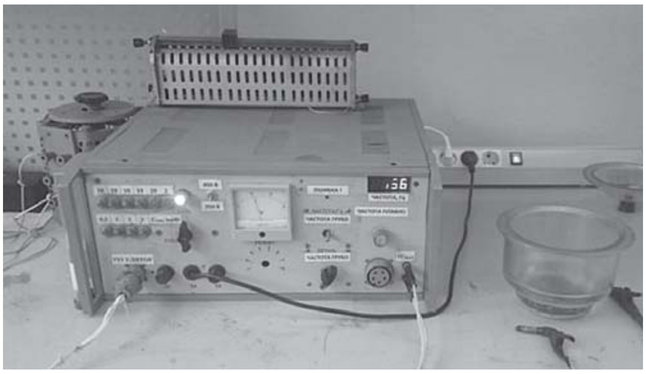

Fig. 1. Electrical discharge dispersing unit

Fig. 2. Dispersion of waste brass in the reactor of the unit

The dispersion process was carried out as follows. Before starting the unit, the electrodes were assembled from waste materials. Then lead brass waste of LS58-3 grade ($CuZn_{39}Pb_3$, GOST 15527–2004) in the form of scrap was loaded into the reactor and the dielectric fluid — isopropyl alcohol — was poured into the reactor (GOST 9805–84) (**Fig. 2**).

On the control panel of the unit we adjusted technological parameters: capacitance of discharge capacitors and pulse repetition rate. Then, using a voltage regulator, the voltage was set to a value at which the electrical breakdown of the dielectric fluid in the interelectrode space occurred. During the formation of the discharge channel, pieces of metal waste at the discharge point melted and vaporized. The dielectric fluid in the electric discharge channel also boiled and vaporized, forming a gas bubble. Droplets of molten and vaporized metal waste fell into the liquid working medium with formation of spherical and elliptical particles, as well as agglomerates. During the operation of the unit, the following parameters of the electroleroson dispersion process were controlled: the level of the dielectric fluid, as well as the behavior of spark formation and the temperature of the coolant. If necessary, the power of spark discharges was regulated by changing the operating modes of the unit.

Mathematical modeling of the process of electrodispersing of lead brass was carried out by setting a, where as factors influencing the optimization parameter (average particle size) were chosen variable parameters of the unit operation: electrode voltage, pulse repetition rate and capacitance of discharge capacitors. The values of selected levels of varying factors are given in **Table 1**.

Table 1
Intervals and levels of factor variation

Levels of factor variation	Code designation	U, V	γ, Hz	C, μF
		X_1	X_2	X_3
Base level	0	150	75	45
Variability interval	Δx_i	50	25	20
High level	+1	200	100	65
Low level	–1	100	50	25

Reaching the optimal value of the average particle size was carried out by the steepest ascent method of Box and Wilson. The optimization task was confined to the experimental determination of such a combination of factor levels, at which the maximum (minimum) value of the output parameter is achieved.

X-ray studies of composition, structure and properties of particles were carried out on modern equipment.

Photographs of the powder particle shape were obtained using a "Quanta 600 FEG" electron-ion scanning microscope. Size analysis was performed on a laser particle size analyzer "Analysette 22 NanoTec". *X*-ray microanalysis of powders was carried out on the energy dispersive *X*-ray analyzer of the company "EDAX" (Netherlands), integrated into a scanning electron microscope "QUANTA 200 3D" (Netherlands). *X*-ray diffraction analysis of powders was performed on an *X*-ray diffractometer "Rigaku Ultima IV" (Japan).

Results of the research

The main technological parameters of the dispersing process are electrode voltage, pulse repetition rate and capacitance of discharge capacitors. Varying these parameters influences the mass and quantitative productivity of the electric discharge dispersing unit, which is directly related to the particle size of the obtained powder. Therefore, these parameters were chosen as factors influencing the optimization parameter — average particle size.

According to the calculations, the regression equation for the mathematical description of lead brass waste dispersion process in isopropyl alcohol was obtained.

$$\hat{y} = 12 + 5.9X_1 + 1.7X_2 + 2.3X_3 + 0.61X_1X_2 + \\ + 0.76X_1X_3 + 0.54X_1X_2X_3 , \quad (1)$$

where X_1, X_2, X_3 — coded values of factors (electrode voltage, pulse repetition rate and discharge capacitor capacity, respectively); $\hat{y}$ — optimization parameter (average particle size, μm).

As we can see from the equation, the greatest influence on the optimization parameter is exerted by the voltage at the electrodes and the capacitance of the discharge capacitors (the factors have the largest coefficients). The resulting equation was used to calculate the steepest ascent

Table 2
Steepest ascent calculation

Item	X_1 (U, V)	X_2 (γ, Hz)	X_3 (C, μF)	$\hat{y}$, μm
Base level	150	75	45	–
b_i coefficient	5.9	1.7	2,3	–
Variability interval ξ_i	50	25	20	–
$b_i \cdot \xi_i$	295	42.5	46	–
Step Δ_i	14.75	2.13	2.3	–
Rounded step	15	3	3	–
Test 1	165	78	48.0	14.3
Test 2	180	81	51	16.88
Test 3	195	84	54	19.52
Test 4	200	87	57	21.0
Test 5	200	90	60	21.8
Test 6	200	93	63	22.6
Test 7	200	96	65	23.3
Test 8	200	99	65	23.6
Test 9 (max)	200	100	65	23.8

of the response surface. The steepest ascent was started from the zero points (base levels) (**Table 2**).

According to the calculations, the limiting value of the optimization parameter $\hat{y}$ (average particle size) was determined, which was: 23.8 μm at an electrode voltage of 200 V, pulse repetition rate of 100 Hz and discharge capacitor capacity of 65 μF.

Further studies of composition and structure of powder particles were carried out at optimum dispersing modes.

The dispersion medium (isopropyl alcohol) has physical, chemical, detergent and mechanical effects on the process and products of electrical erosion. It provides high technological indicators of electrodispersing, thermal stability of physical and chemical properties under the influence of electric discharges and high cooling capacity. Isopropyl alcohol, being a hydrocarbon liquid, influences the chemical and phase composition of erosion products. Various chemical compounds are formed during chemical interaction with them.

When the electric discharge passes through, decomposition of the dielectric fluid takes place. In the process of dispersion erosion products — metal waste particles — are ejected in molten form from the electric discharge channel into the reactor filled with dielectric fluid and crystallize very quickly. The process of rapid crystallization of the molten material in the liquid working medium promotes the formation of sphere- and ellipse-shaped particles. The results of morphology study of the obtained powders are presented in **Fig. 3**. It is shown that the particles have mainly spherical and elliptical shapes, as well as agglomerates formed as a result of adhesion of particles close in temperature.

Analysis of the powder particle size distribution performed with the Analysette 22 NanoTec particle size analyzer showed that the EDM particles can range in size from 0.5 μm to 100 μm with two pronounced peaks of

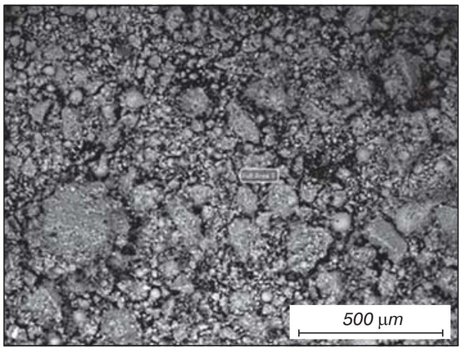

Fig. 3. Powder particle morphology

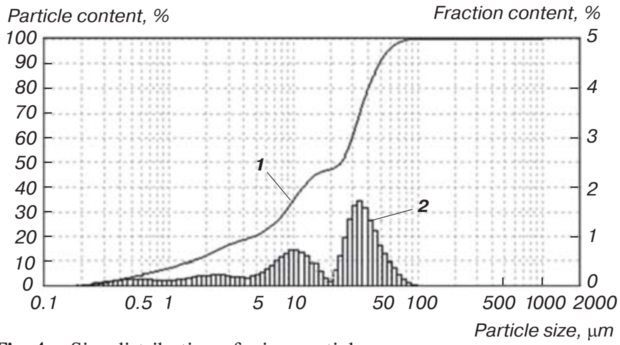

Fig. 4. Size distribution of microparticles:
1 — integral curve; *2* — histogram

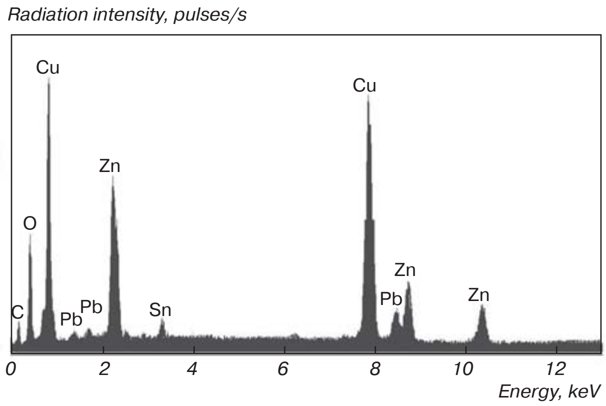

Fig. 5. Spectrogram of powder particles elemental composition

10 μm and 35 μm. In the dispersion process, smaller particles are formed by crystallization from the vapor phase and larger particles from the melt phase. The average particle size is 23.8 μm (**Fig. 4**).

The analysis of elemental composition has established that free carbon is contained on the surface of powder particles. Other chemical elements Cu, Zn, Pb, Sn are distributed relatively uniformly. The presence of free carbon is due to the chemical composition of the dispersion medium, which is carbon-containing (**Fig. 5**) and allows to expand the field of application of the obtained charge, in particular, in the manufacture of antifriction coatings.

The involvement of oxygen molecules within isopropyl alcohol in the reaction zone, the melt zone, led to the formation of various metal oxides. *X*-ray diffraction analysis of the obtained powders showed the presence of phases

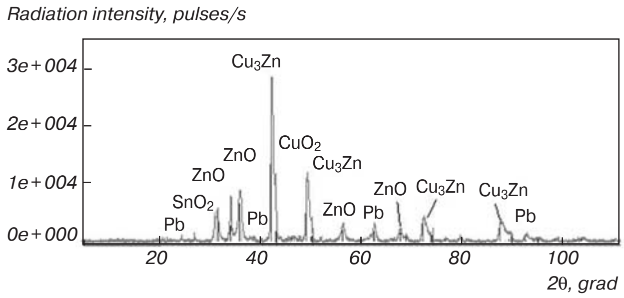

Fig. 6. Diffractogram of powder particles phase composition

Cu_3Zn, Pb, ZnO, CuO_2, SnO_2. There are no carbide-forming elements in the alloy composition (**Fig. 6**).

Electrical discharge dispersion is a cost-effective method of powder production, as it allows the use of waste as raw material. Due to the good filtration of the dielectric fluid, it can be reused until it is completely used up. Due to this, lead brass powders obtained as a result of electrical discharge dispersion are 1.3 times cheaper than powders obtained by industrially applied methods.

The obtained research results contribute to the development of further research in the field of powder metallurgy and improvement of technological processes of manufacturing of new coatings and parts based on electroerosive brass powders.

Conclusion

1. The use of technology of electric discharge dispersing for processing of waste brass LS58-3 ($CuZn_{39}Pb_3$) and obtaining powders on its basis is relevant. This method of metal waste recycling allows to obtain charge particles with a given complex of properties, which can be used in the creation of corrosion-resistant, wear-resistant coatings with improved antifriction properties, as well as obtaining alloy blanks by various methods of compacting and sintering. The formation of spherical-shaped particles in the process of electrodispersion of waste brass is a very important feature of this process, which allows us to recommend it for use in additive technologies, where sphericity is the main requirement for powders for additive machines.

2. Setting of the full factor experiment allowed to mathematically model the process of dispersion of brass waste and optimize it by determining the optimal modes of powder production.

3. The analysis of the results of the conducted studies showed that the physicochemical properties of the charge dispersed by electrical erosion of brass LS58-3 ($CuZn_{39}Pb_3$) are influenced by the chemical composition of the dielectric fluid, as well as dispersion modes. By varying the technological parameters of the unit, it is possible to produce powder of different size and shape. Carburization of particle surface due to the process of electroerosion in isopropyl alcohol has a favorable effect on antifriction properties of further products from the obtained powders.

4. The use of electric discharge dispersing technology increased the economic efficiency of metal waste recycling due to low energy consumption of the powder production process.

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