Composition, structure, and properties of heat-resistant alloys samples made from powders obtained by electroerosion of waste nickel alloys in kerosene

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In the work scientific and applied principles of conjugation of the technology of obtaining nickel powder materials from waste heat-resistant nickel alloy of ZhS6U (ЖС6У) grade by electroerosion dispersion and technology of their compacting by spark plasma sintering are implemented.
The purpose of this work was to study the composition, structure, and properties of heat-resistant products made by spark plasma sintering of powders obtained by electrodispersion of ZhS6U alloy waste in illuminating kerosene.
Electrodispersion of waste heat-resistant ZhS6U alloy was carried out in illuminating kerosene on a patented unit. The conducted studies have shown that the method of electroerosion dispersion of ZhS6U alloy waste in illuminating kerosene makes it possible to obtain heat-resistant nickel alloy powder with a uniform distribution of alloying elements.
The obtained heat-resistant nickel powder was sintered in the SPS 25-10 "Thermal Technology" system (USA). The economic efficiency of this method is due to the use of waste ZhS6U grade alloy in a low-energy production technology of heat-resistant alloys. The implementation of the planned activities will solve the problem of recycling waste heat-resistant nickel alloys and their reuse in the manufacture of heat-resistant products.
Based on the experimental studies aimed at investigating the composition, structure and properties of sintered samples of heat-resistant alloys, from electric erosion powders obtained in illuminating kerosene, a high efficiency of spark plasma sintering technology has been shown, which provides for suppression of grain growth, low porosity and high physical and mechanical properties with uniform heat distribution over the sample and a short working cycle time. It is noted that the heat resistant alloys made of particles of ZhS6U alloy dispersed by electric erosion and obtained by spark plasma sintering have the following characteristics: has fine grain structure in the absence of pores, cracks and other defects; the main elements in the alloys are: C, Al, W, Cr, Fe, Mo, Ni, Nb, Co и Ti; main phases: Ni, $Ni_{17}W_3$, C_5NbTi_2 and $Al_{0.9}Ni_{4.22}$; porosity is 0.18%; average microhardness value is 460 $HV_{0.2}$.

Key words: ZhS6U grade alloy waste, electroerosion dispersion, powder, spark plasma sintering, heat-resistant nickel alloy, properties.

DOI: 10.17580/nfm.2023.01.05

Introduction

The gas turbine engine (GTE) industry is developing by increasing the operating temperature of gas at the turbine inlet, which imposes higher requirements for the quality of heat-resistant alloys, such as ZhS6U [1–4]. On the other hand, the presence of expensive alloying components in their composition requires reducing their cost. One of the popular solutions in this direction is the widespread recycling of heat-resistant alloys for the production of GTE parts, in particular blades, with the recommended recycling rate of up to 100% [5–8].

Two solutions to this problem are proposed. The first one is the milling of zhS6U alloy waste by electroerosion dispersion in liquid dielectrics. Powder materials obtained by electroerosion method have mainly spherical particle shape [9–12]. The second is the use of the spark plasma sintering method to produce a heat-resistant nickel alloy from powder obtained by electroerosion dispersion of the ZhS6U alloy in illuminating kerosene. Obtaining a heat-resistant alloy by spark plasma sintering under conditions of rapid heating and a short duration of the working cycle should help to improve its physical and mechanical properties by suppressing grain growth and obtaining an equilibrium state with submicron and nanoscale grains [13–15].

To date, in the modern scientific and technical literature there is no complete information about the use of particles of ZhS6U alloy dispersed by electro-erosion as a charge for the production of heat-resistant nickel alloys. For these purposes, a comprehensive theoretical and experimental research is required.

The purpose of this work was to study the composition, structure, and properties of samples of heat-resistant alloys produced by spark plasma sintering of powders obtained by electrodispersion of ZhS6U grade alloy waste in illuminating kerosene.

Materials and methods of research

To carry out the planned research, metal wastes of ZhS6U alloy were selected, namely spent blades of GTE. The chemical composition of the alloy is shown in **Table 1**.

Table 1
Chemical composition of the ZhS

Alloy	Content of elements, wt.%									
ZhS6U	C	Cr	Co	Mo	W	Nb	Al	Ti	Zr	B
	0.13–0.2	8.0–9.5	9.0–10.5	1.2–2.4	9.5–11.0	0.8–1.2	5.1–6.0	2.0–2.9	up to 0.04	up to 0.035
	Fe	Si	Mn	S	P	Ce	Pb	Bi	Y	Ni
	up to 1.0	up to 0.4	up to 0.4	up to 0.01	up to 0.015	up to 0.02	up to 0.001	up to 0.0005	up to 0.01	other

As a working fluid we used the most technologically advanced, affordable and cheap carbon-containing liquid – illuminating kerosene (TI No. 38.401-58-10-01).

The methodology for obtaining powders by electrodispersion of zhS6U alloy wastes in illuminating kerosene is presented below [9–12] **(Fig. 1)**. In the beginning, the assembly of electrodes (*5* and *6*) from dispersible metal waste (*8*) is performed. Then pellets of dispersible metal waste (*8*) are loaded into the reactor (*3*) and the working fluid (*10*) is poured. On the control panel of the pulse generator (*2*) the required parameters for electrodispersing of metal waste are setting: capacitance of discharge capacitors and pulse repetition rate. Then by using the voltage regulator (*1*) such voltage is set, at which there is an electrical breakdown of the working fluid (*10*), which is in the interelectrode space. When the discharge channel is formed, pieces of waste metal at the discharge point melt and evaporate. The working fluid (*10*) in the electric discharge channel also boils and evaporates, forming a gas bubble (*9*). Droplets of molten and evaporating waste metal fall into the working fluid environment to form spherical and elliptical particles (*7*), as well as agglomerates. Shaker (*4*) moves one of the electrodes and provides a continuous process of electrodispersion.

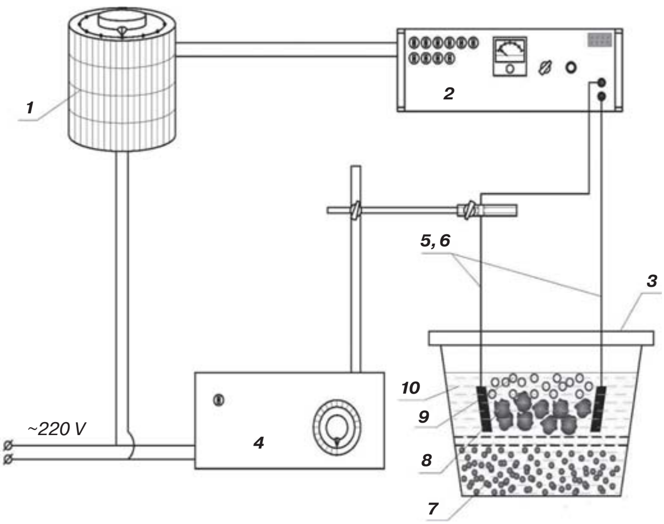


Fig. 1. Schematic diagram of an electrodispersing unit for metal waste: *1* – voltage regulator; *2* – pulse generator; *3* – reactor; *4* – shaker; *5*, *6* – electrodes; *7* – electroerosion particles; *8* – metal waste; *9* – gas bubbles; *10* – working fluid

The following electrical parameters were used: electrode voltage of 130...150 V; the capacity of condensers was 55...60 μF; pulse frequency 260...300 Hz. The electroerosion nickel powder obtained was examined by various methods.

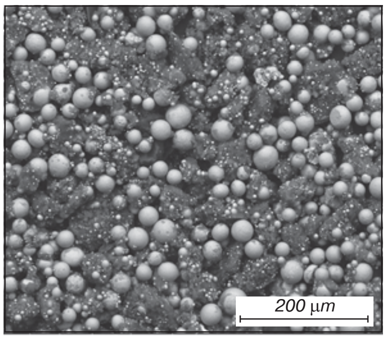


Fig. 2. Raster electron microscopic image of erosion powders

Microanalysis of powder particles, carried out with a scanning electron microscope "QUANTA 600 FEG", showed that the powder, obtained by the EED method from waste alloy ZhS6U, consists mainly of particles of spherical shape and agglomerates **(Fig. 2)**.

Analysis of the particle size distribution of the powder obtained with a particle size analyzer "Analysette 22 NanoTec" showed that the powder particles have sizes from 0.5 to 100 μm with two pronounced peaks at 10 μm and 50 μm. *X*-ray microanalysis of powder particles, performed with the EDAX *X*-ray energy dispersive analyzer, integrated into the QUANTA 600 FEG scanning electron microscope, showed that in the powder obtained by electroerosion from the ZhS6U alloy waste, carbon is found on the surface of disperse particles, and all other elements such as Cr, Fe, Co, Ni, Nb, Mo, Ti, Al and W are relatively evenly distributed.

The conducted studies have shown that the method of electroerosion dispersion of ZhS6U alloy waste in illuminating kerosene makes it possible to obtain heat-resistant nickel alloy powder with a uniform distribution of alloying elements.

Sintering of heat-resistant nickel powder was carried out in the SPS 25-10 "Thermal Technology" system (USA) at temperature $T = 1300$ °C, pressure $P = 40$ MPa, and holding time $t = 10$ min.

The microstructure of the alloy was examined on an electron-ion scanning (raster) microscope with field emission of electrons "QUANTA 600 FEG" (Netherlands).

X-ray spectral microanalysis of the alloy was carried out on an energy dispersive *X*-ray analyzer by "EDAX" (Netherlands), integrated in a scanning electron microscope "QUANTA 200 3D" (Netherlands).

Phase analysis of the alloy was performed on an *X*-ray diffractometer "Rigaku Ultima IV" (Japan).

The alloy porosity was determined using an Olympus GX51 optical inverted microscope with image quantitative analysis software. The prepared samples had no marks of grinding, polishing or pitting of structural components.

Microhardness of an alloy on a surface was determined by means of the automatic system of microhardness analysis AFFRI DM-8 by the method of micro-Vickers at loading on an indenter of 200 g on ten prints with free choice of a place of a prick according to GOST 9450–76 (Measurement of microhardness by indentation of diamond tips). The indenter holding time was 10 s.

Results

Analysis of the microstructure of a heat-resistant alloy made of nickel powder, showed that it has a fine-grained structure in the absence of pores, cracks and other defects **(Fig. 3)**.

Summarized data on results of microstructure study and electron probe microanalysis (EMPA) of sintered samples, made of electro-erosion powders obtained in kerosene, are presented in **Fig. 4.** Each chemical element on the spectra corresponds to a peak of a certain height.

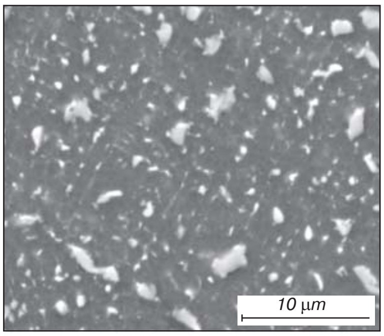
Based on the analysis of spectrograms of elemental composition it was found that the alloy consists of the following elements evenly distributed over the volume: C, Al, W, Cr, Fe, Mo, Ni, Nb, Co, and Ti. It is noted that the elemental composition of the sintered high-temperature alloy practically coincides with the elemental composition of the initial electroerosion powder.

The results of the *X*-ray structure (phase) composition of sintered samples are shown in **Fig. 5.** The analysis of diffractograms of the phase composition of the studied alloys showed the presence in them the following phases: intermetallic $Ni_{17}W_3$, C_5NbTi_2, $Al_{0.9}Ni_{4.22}$ and pure Ni.

The results of sintered heat-resistant alloys samples porosity investigation by metallographic method using an optical microscope OLYMPUS GX51 are shown in **Fig. 6** and in **Table 2**.

As a result of the study of the poroџsity of sintered samples by metallographic method, it was found that its value is about 0.18%. The low porosity values of the obtained samples are due to the effect of the spark discharge plasma during sintering process.

Fig. 3. Alloy microstructure



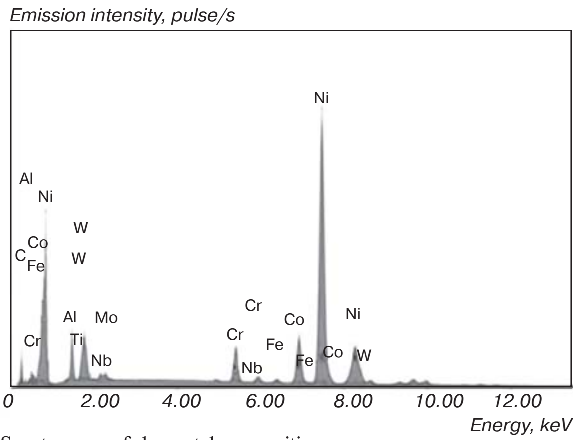


Fig. 4. Spectrogram of elemental composition

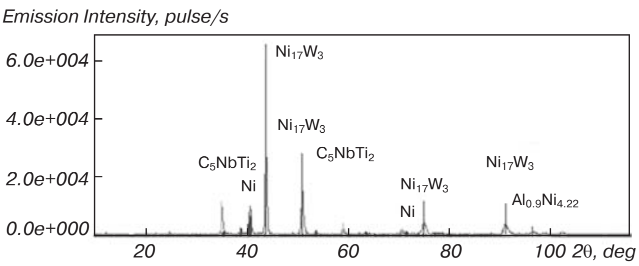


Рис. 5. Phase composition diffractogram

Table 2

Sintered alloy porosity parame

Analysis surface area, μm^2	Porosity, %	Pore size, μm		
		minimum	maximum	average
218456.8	0.18	0.2	0.4	0.3

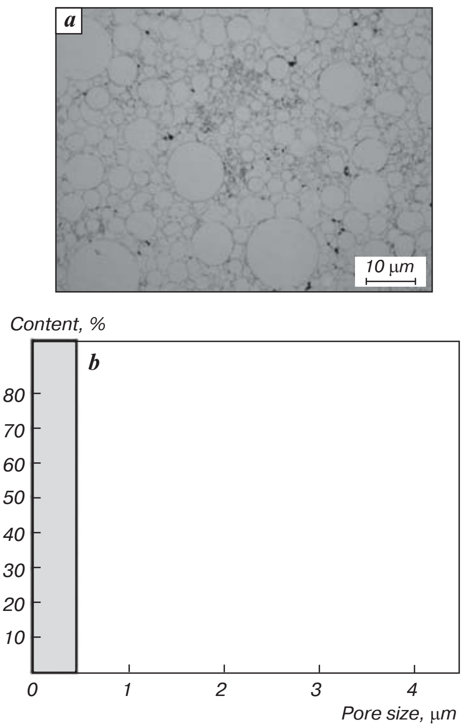


Fig. 6. Results of the alloy porosity study:
a — microstructure of the cross-sectional profile of the sample,
b — histogram of pore size distribution

It was established experimentally that the microhardness of sintered samples made by spark plasma sintering of ZhS6U alloy particles obtained by elctroerosion dispersion method is 460 $HV_{0.2}$ with a 3σ confidence interval equal to ±144.5.

Fine grain and the presence of a large number of alloying elements Al, W, Cr, Fe, Mo, Ni, Nb, Co, Ti in sintered samples of heat-resistant alloys should provide them with high heat resistance and ductility.

This work solves an important scientific and practical problem aimed at creating an advanced, environmentally friendly, low-tonnage and waste-free technology for producing new electroerosion nickel powders and alloys based on them that are suitable for industrial application.

In the work scientific and applied principles of coupling technology of obtaining new powder materials from waste heat-resistant alloys of ZhS6U grade made by electroerosion dispersion and technology of their compacting by spark plasma sintering are implemented.

The above confirms the necessity and relevance of developing scientific principles and technological foundations for the recycling of heat-resistant alloys by electroerosion dispersion of its waste, followed by spark plasma sintering.

Conclusion

1. On the basis of the experimental studies aimed at investigating the composition, structure and properties of sintered samples of heat-resistant alloys, made from electric erosion powders obtained in illuminating kerosene, the high efficiency of spark plasma sintering technology, which provides for suppression of grain growth, low porosity and high physical and mechanical properties with uniform heat distribution over the sample and a short working cycle time, is shown.

2. It is noted that heat-resistant alloys obtained by spark plasma sintering from particles of the ZhS6U alloy made by electroerosion dispersion have the following characteristics:

– ine-grained structure without pores, cracks and other defects;

– main elements in the alloys are: C, Al, W, Cr, Fe, Mo, Ni, Nb, Co and Ti;

– main phases: Ni, $Ni_{17}W_3$, C_5NbTi_2 and $Al_{0.9}Ni_{4.22}$;

– porosity is 0.18%;

– average value of microhardness is 460 $HV_{0.2.}$

3. The economic efficiency of this method is due to the use of waste ZhS6U grade alloy in a low-energy production technology of heat-resistant alloys. The implementation of the planned activities will solve the problem of recycling waste heat-resistant nickel alloys and their reuse in the manufacture of heat-resistant products.

Acknowledgments

The work was carried out with the financial support of the grant of President of the Russian Federation (НШ-596.2022.4).

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