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PRODUCTION OF POWDER OF THE Ni-Cr-Al-Y SYSTEM ALLOY DOPED WITH SILICON BY THE POWDER METALLURGY METHOD

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The influence of solid-phase interaction of Ni–Cr–Al–Y alloy with silicon at up to 1100 °C temperature was studied. It was established that to produce the Ni–Cr–Al–Y + Si alloy it is expedient to use nickel mechanically doped with silicon as one of the initial intermetallic components of the alloy, this preventing presence of free silicon in the alloy and ensuring uniform distribution of silicon through the powder volume.

Keywords: detonation spraying, powders, heat-resistant alloy Ni-Cr-Al-Y, mechanical doping with silicon, solid-phase interaction, phase composition, distribution of doping elements

Heat-resistant nickel-base alloys are widely applied to manufacture parts operating under extreme conditions of high temperatures and aggressive environments [1]. Development of new materials for protective coatings by adding doping elements to compositions of standard alloys in order to improve their service characteristics, such as heat and corrosion resistance, is of high current importance.

According to the diagram of dependence of heat and corrosion resistance of coatings upon the content of chromium in them [2], the chosen alloy for the investigations (composition, wt.%: 79 Ni, 15 Cr, 5.8 Al, 0.2 Y) is classed with the most heat-resistant ones, and doping with active additions is one of the ways of increasing its functional characteristics of heat and corrosion resistance. Silicon is not often referred to as belonging to the doping elements (boron, magnesium, zirconium, hafnium, etc.), although it should play an important role as a coating element that forms a strong and dense self-passivating oxide film during oxidation. Some researchers studied the effect of silicon on resistance of alloys at increased temperatures [3, 4]. However, these studies are of a contradictory character and contain no generalisations on the protection mechanism of the coatings to be used as a basis to select certain amounts of doping additions or compounds containing the required elements and set the method for adding them to an alloy.

The purpose of this study was to investigate solidphase interaction of initial components of the Ni-Cr-Al–Y alloys with silicon within a service temperature range (up to 1100 °C). The alloys were produced by the powder metallurgy method. Several methods are available for adding doping impurities [5]. Silicon can be added to the initial mixture of nickel, chromium and aluminium powders, followed by powder metallurgy operations (mixing, crushing or mechanical activation, heat treatment, etc.), or it is possible to first produce a silicon compound with one or several initial components and then mix it with other components of the alloy. The doping method is determined by technological peculiarities of subsequent processes. This study considered several methods for adding silicon.

One of methods for adding a silicon impurity to a complex nickel-base alloy is adding it together with yttrium oxide during the process of production of a standard alloy powder. Yttrium oxide and silicon (up to 4 wt.%) are added at a stage of mixing to a mixture of Ni₃Al and Cr(Ni) powders preliminarily produced by solid-phase synthesis in vacuum. Then, following the flow diagram of the process developed by the authors, the diffusion processes of interaction of silicon with main phases of the alloy, i.e. Ni₃Al and Cr(Ni), should take place during vacuum heat treatment.

The interaction products after vacuum heat treatment at a temperature of 1000 $^{\circ}$ C for 2 h were investigated by X-ray analysis and scanning electron mi-



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SCIENTIFIC AND TECHNICAL



Figure 1. Microstructure of particle of IPM synthesised powder (a) and distribution of silicon in it (in reflected electrons) (b)

croscopy. According to the X-ray analysis data, the synthesised material consists of a mixture of the main phases, i.e. Ni_3Al and Cr(Ni), and silicon, which is in a free state. The data of scanning electron microscopy and energy-dispersive method given in Figure 1 and in Table 1 are indicative of a non-uniform distribution of silicon through the volume. It can be seen that the maximal amount of silicon is concentrated on surfaces of the particle.

Elemental composition of the produced powder is as close as possible to that of the chosen standard powder.

Investigations show that adding the doping impurity at a stage of mixing of the preliminarily synthesised powders of Ni₃Al, Ni(Cr) and Y₂O₃, followed by heat treatment in vacuum at a temperature of 1000 °C, does not provide completeness of the diffusion processes, and does not yield the products containing no free silicon. This gives no way of fully



Figure 2. Diffraction patterns of mechanical synthesis products of the Ni–Si system as a function of weight ratio at process duration of 60 min: 1 - 1:5; 2 - 1:10; 3 - 1:20



Table 1. Chemical composition of powder of Ni-Cr-Al-Y alloy,

reproducing the elemental composition of a material in detonation sprayed coatings due to fractionation. Because of the above-said, as well as considering the data on substantial solubility of silicon in nickel (5%) at room temperature to form solid solution, it was expedient to use nickel doped with silicon during mechanical synthesis as one of the initial components of the alloy. In study [6] this solution is identified as α -solution, and in study [7] — as γ -solution. In our investigations we designate it as γ -Ni, in analogy with solid solutions of the austenitic class.

Investigations of the process of solid-phase interaction of nickel and silicon in mechanical synthesis (mechanical doping) included evaluation of the process on the basis of technological factors, such as weight ratio (ratio of powder weight to weight of milling bodies) and duration of the process.

To study mechanical synthesis, the nickel and silicon powders (in amounts to yield 4 wt.% Si in nickel) were preliminarily mixed in a planetary-type mill in the reversal mode for 2 h in alcohol to homogenise the reaction mixture. After drying, the mixture was subjected to high-energy treatment.

Mechanical synthesis is a high-energy crushing that induces internal stresses in a solid body, this causing deformation of atomic bonds, formation of defects of a crystalline structure and excitation of electron sub-system of a crystal. At that, each particular case is characterised by predominance of certain channels of relaxation of the stored energy. Thus, in formation of new boundary surfaces the energy consumption for restructuring is minimal, and atoms in the surface layer have a high margin of the excessive energy, this creating favourable conditions for chemical transformations of contacting materials.

 Table 2. Characteristics of mechanical synthesis products of Ni–Si system

Weight ratio — powder to spheres weight ratio	Duration, min	Phase composition	Lattice spacing of nickel, nm	
1:5	30	Ni, Si	0.352	
	60	Ni, Si	0.353	
1:10	30	Ni, Si	0.352	
	60	γ-Ni, Si	0.350	
1:20	30	γ-Ni, Si	0.349	
	60	γ-Ni	0.347	





Figure 3. Morphology of powders produced by IPM (a) and «Stark» Company (b)

The process was carried out in a pulse mode, which led to a change in the crystalline lattice energy due to formation of different structural defects (dislocations, vacancies). This resulted in stimulation of the diffusion processes in the system by the stored energy at certain technological parameters.

The mechanical synthesis products were examined by X-ray phase analysis (Table 2, Figure 2).

Analysis of the obtained experimental data indicates to a substantial effect of the energy intensity of the process, which is a function of the technological parameters at a constant speed of rotation of the reactor. At a weight ratio of 1:5, the milling products are a mixture of initial components, independently of the process duration.

A slight change in the character of diffraction maxima of nickel and silicon is indicative of occurrence of the first stage of deformation of the powder (Figure 2, curve t). Increase of weight ratio to 1:10 is accompanied by partial amorphisation of silicon, this being evidenced by lowering and broadening of its diffraction maxima. Increase in imperfection and stress level of nickel shows up in broadening at high angles of its diffraction images (Figure 2, curve 2). Increase of the process duration to 60 min leads to the beginning of formation of solid solution of silicon in nickel (γ -Ni), this being evidenced by a change in the lattice spacing of nickel.

A weight ratio of 1:20 is accompanied by an even more pronounced intensification of the process. According to the X-ray analysis data, not only the quantitative composition of the interaction products changes, but also a distortion of the pattern of diffraction maxima of γ -Ni takes place, which is indicative of a stressed and non-equilibrium state of the system

Table 3.	Chemical	composition	of	powders,	wt.%
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Manufacturer of powder	0	Al	Si	Cr	Ni	Y
IPM	0.15	5.38	2.6	15.40	76.26	0.21
«Stark» Company	_	9.54	-	21.91	67.73	0.82







Figure 5. Microstructure of particles of the powder of mechanically synthesised alloy Ni–Cr–Al–Y (a) and distribution of silicon in them (in reflected electrons) (b)



SCIENTIFIC AND TECHNICAL

(Figure 2, curve 3). At the 60 min duration of the process, the product of mechanical synthesis is solid solution of silicon in nickel.

Therefore, the investigations conducted made it possible to identify the technological parameters of mechanical synthesis, the product of which is a powder of solid solution of silicon in nickel (γ -Ni).

According to the technological process, production of the Ni–Cr–Al–Y + Si alloy powder provides for the use of individual intermetallics Ni₃Al and Ni(Cr) after the low-temperature solid-phase synthesis, followed by their mixing with yttrium oxide, granulation and vacuum heat treatment. The mechanically synthesised powder of solid solution of silicon in nickel is used as one of the initial components at a stage of production of the intermetallic powders. The special consideration was given to investigation of the effect of doped nickel on temperature-time parameters of synthesis of intermetallic phases Ni₃Al and Ni(Cr).

The data of X-ray analysis of intermetallics Ni_3Al and Ni(Cr) synthesised in vacuum, which make the base of the Ni–Cr–Al–Y + Si alloy, indicate to the absence of free silicon in them.

The phase and elemental composition, as well as morphology of the produced powder were determined by the methods of X-ray analysis and scanning electron microscopy with energy-dispersive analysis.

As can be seen from Figure 3, a, the produced powder consists of equiaxed particles with a mean diameter of about 60 μ m, which meet requirements to the detonation spraying powders.

X-ray microanalysis of the experimental powder (Figure 4, *a*; Table 3) confirmed the presence of silicon in it. Energy-dispersive scanning of the produced pow-

der of the Ni–Cr–Al–Y alloy doped with silicon reveals a non-uniform distribution of all elements through the volume (Figure 5).

Therefore, the work done to evaluate solid-phase interaction of initial components of the Ni-Cr-Al-Y alloy with silicon in a service temperature range (up to 1100 °C) made it possible to establish that direct doping of intermetallic components Ni₃Al and Ni(Cr) of the Ni–Cr–Al–Y alloy with silicon in vacuum heat treatment at a temperature of 1100 °C leads to the undesirable presence of free silicon in the solid-phase interaction products, as well as to the non-uniform distribution of silicon in the alloy. To produce the Ni-Cr-Al-Y-Si alloy, it is expedient to use nickel mechanically doped with silicon as one of the initial intermetallic components of the alloy, since this allows avoiding the undesirable presence of free silicon in the alloy and ensures the uniform distribution of silicon through the powder volume.

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NEWS

EFFECTIVE APPLICATION OF LASER WELDING

At Chelyabinsk pipe rolling plant (OJSC «ChTPZ», Chelyabinsk region) a pilot welding of pipes of 530×8 mm in size with use of a laser equipment was performed. The pipes were welded using two lasers of 8.3 kW total capacity with coincidence of two beams into one welding pool. Welding was performed using a filler wire Sv-08G2S of 1.2 mm diameter. Two pipe samples, welded by laser, were tested at the test grounds by supply of hydraulic pressure until fracture. The pipe fracture occurred at pressure of 203 atm: the weld withstood the pressure of 200 atm.

