Structure and properties of the composite material based on heat-resisting VKNA-4U alloy with oxide stiffening fillers, designed for additive manufacturing technologies of power fasteners for gas turbine engines (GTE)

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Abstract. The influence of the structure and concentration of the stiffening component on the physicomechanical properties of the heat-resisting intermetallic alloy VKNA-4U was investigated. The dependence of the strength properties of the alloy on the composition and ratio of the hardeners α-Al₂O₃, Y₂O₃, HfO₂, ZrO₂ was experimentally determined. Using scanning electron microscopy (SEM), it was found that when the alloy is reinforced with hafnium, yttrium and aluminum oxides, the dispersed hardener does not dissolve in the matrix. These compositions are considered as the most promising for the stiffening of VKNA-4U alloy.

It is shown in this paper that the required content of the oxide reinforcing filler in the VKNA-4U matrix is 2%, at which a balance is reached between the maximum value of strength and ductility. In the future, VKNA-4U powder composition mechanically alloyed with α-Al₂O₃, Y₂O₃, HfO₂ oxides can be used as a building material for additive synthesis technologies for power fasteners of gas turbine engines (GTE).

1. Introduction
One of the most important tasks of modern materials science is the creation of materials with high operating temperature while maintaining a low density, due to the need to increase the temperature of the gas at the turbine inlet. This can be achieved by developing a new class of high-temperature materials or applying new principles of hardening metal heat-resistant matrix, which will allow to obtain working temperatures higher than that of existing nickel alloys.

Stiffening and strengthening mechanisms, which are based on a multi-component doping, inevitably lead to a decrease in the melting temperature (Tm), and therefore limit the upper limit of operating temperatures. One of these principles is implemented in metal-ceramic composite materials, in which the hardening phase consists of ultrafine particles artificially introduced into the matrix, high-strength, thermodynamically stable, not soluble and not interacting with the matrix material throughout the temperature range up to the melting point of the matrix alloy. The hardening effect of the introduction of ultrafine particles is summed up with the effect of using traditional hardening mechanisms, but, unlike the latter, it is maintained at extremely high temperatures when other hardening mechanisms cease to operate [1, 2].

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For power fasteners of gas turbine engines, it is advisable to use alloys with a working temperature equal to the maximum temperature of their installation. For combustion chambers, high-pressure boiler and turbine the alloys are needed with operating temperatures up to 1200°C. In most serial domestic engines power fasteners are made of serial deformable nickel alloys such as ЭИ437БУ, ЭИ698, ЭП742. The maximum operating temperature of these alloys is 750°C, above which the material softens and its strength properties sharply decrease [3, 4].

FSUE “VIAM” has developed a number of high-strength and heat-resistant alloys that can be used to manufacture parts of an aircraft. For example, (Ni3Al) has a number of important advantages over industrial nickel alloys - operating temperature up to 1200°C for a long time (short-term temperature rises up to 1250°C are possible), the maximum heat resistance among alloys of its class at the operating temperature [5]. The disadvantage of all nickel alloys is a decrease in the strength characteristics with increasing operating temperature. This problem can be solved through adding the cermet reinforcing filler to them. It is believed that the enlargement of particles at high temperature occurs as a result of the dissociation of small particles of the following diffusion of components through the matrix and their deposition on larger particles. It follows that the fine phase should have the highest possible thermodynamic stability, and oxides meet these requirements the best [6, 7].

The addition of the oxide filler to the VKNA-4U alloy should increase the heat resistance of the finished material and eliminate the high-temperature creep of the matrix alloy, which will allow to maintain the strength and geometry of the power fasteners of GTE made from this alloy.

The purpose of this paper is to study the effect of oxide reinforcing filler on strength characteristics of matrix material of VKNA-4U alloy at room temperature.

2. Materials and methods of experiment

As a material for studying the effect of reinforcement with nanoscale powders of oxides α-Al2O3, Y2O3+Al2O3, HfO2+Y2O3+Al2O3, HfO2+Y2O3, HfO2+ZrO2+Y2O3, Al2O3+ZrO2+Y2O3, ZrO2+Y2O3 the heat-resisting intermetallic alloy VKNA-4U was selected. The starting components for the manufacture of composite powder based on VKNA-4U alloy were domestic powders.

The initial charge billet of the matrix alloy was obtained in an induction furnace UNK-1. Next, the atomization technology was applied (obtaining a micro-sized powder by spraying the charge stock) on the sixth generation HERMIGA 10/100 unit.

After gas atomization, the powder of the VKNA-4U matrix alloy was divided into fractions 0-56 µm and 56-120 µm on the Analysette 3 Spartan vibrating screen and the sieve (mesh according to GOST 6613) with a mesh size of 56 microns.

To obtain nanoscale oxide granules α-Al2O3, Y2O3+Al2O3, HfO2+Y2O3+Al2O3, HfO2+Y2O3, HfO2+ZrO2+Y2O3, Al2O3+ZrO2+Y2O3, ZrO2+Y2O3 the plasma-chemical method was applied. The fraction was synthesized on the installation of the plasma torch, the design of which was developed at the Institute of Metallurgy and Materials Science. A. A. Baykova RAS [8]. The principle of synthesis of nanoscale oxide particles is based on the interaction of dispersed raw materials with a jet of thermal plasma containing oxygen.

High-temperature chemical reactions occur in the reactor volume, leading to vaporization of the target product, which is then condensed into nanoparticles. Formed nanoparticles are deposited on the walls of the reactor and on the filter, from where they are removed in the receiving collections of the resulting product. Plasma-chemical unit is designed to obtain nanopowders as a result of oxidative processes, as well as processes of thermal decomposition of compounds. Particles of refractory compounds produced in a plasma-chemical installation, are predominantly spherical. The average particle size is no more than 300 nm.

To assess the interaction of the VKNA-4U matrix and oxide fillers, experimental samples of the following compositions were made: VKNA-4U + ZrO2 – Y2O3 – Al2O3, VKNA-4U + ZrO2 – Y2O3, VKNA-4U + HfO2 – Y2O3, VKNA-4U + HfO2 – Y2O3 – Al2O3, VKNA-4U + Y2O3 – Al2O3, VKNA-4U + HfO2 – Y2O3 – ZrO2. Experimental samples were obtained according to the following scheme: powder composite billet was obtained by the method of mechanical alloying in a planetary mill.
PM400, then the powder material was compacted by cold pressing on a hydraulic press. After mechanical alloying, the average size of the composite granules was 60 µm. Compaction granules were compacted on a hydraulic press ДБ2432 with a force of 160 tons. The compacted samples were mechanically processed and annealed in a vacuum electric furnace СНВГ 4/22 at a temperature close to the working one (1200°C) for 1 h [9, 10]. The study of the interaction of the matrix alloy with stiffening filler was carried out after annealing the samples using micro-X-ray spectral analysis using an attachment with a solid-state silicon detector X- maxN 80. The mapping of the distribution of elements, the visualization of the energydispersion spectra and the calculations of the local composition were carried out using AzTec software modules.

The physicomechanical properties of samples of materials in which the dissolution of the stiffening filler in the matrix alloy did not occur were investigated.

Impact toughness was determined on the installation InstronCEAST 9050, density – as per GOST 18898-89. All tests were carried out at room temperature.

3. Research results and discussion

Due to the fact that the VKNA-4U alloy is an intermetallic alloy, it has specific disadvantages, such as insufficient low-temperature plasticity due to poorly developed mechanism for transferring the deformation along the grain boundaries. From reference sources it is known that with increasing proportion of stiffening component hardness and modulus of elasticity increase, however, at the same time, low-temperature plasticity decreases, so the optimal proportion of stiffening filler was chosen as 2% [11].

Fig. 1.2 shows the results of X-ray spectral analysis of VKNA-4U samples from 2% content of reinforcing fillers.

Figure 1 clearly shows that in VKNA-4U + ZrO2 – Y2O3 – Al2O3, VKNA-4U samples + ZrO2 – Y2O3, VKNA-4U + HfO2 – Y2O3, VKNA-4U + HfO2 – Y2O3 – ZrO2 there is a zone of interaction between the matrix and the stiffening component. Figure 2 shows the result of chemical analysis of a sample with an interaction zone.

![Figure 1. Microstructure of VKNA-4U alloy samples reinforced with various oxides: a - Y2O3](image_url)
- $\text{Al}_2\text{O}_3$ – $\text{ZrO}_2$, $\delta$ - $\text{HfO}_2$ – $\text{ZrO}_2$ – $\text{Y}_2\text{O}_3$, $\sigma$ - $\text{ZrO}_2$ – $\text{Y}_2\text{O}_3$, $\varepsilon$ - $\text{HfO}_2$ – $\text{Y}_2\text{O}_3$, $\phi$ - $\text{Y}_2\text{O}_3$ – $\text{Al}_2\text{O}_3$, $\epsilon$ - $\text{HfO}_2$ – $\text{Y}_2\text{O}_3$ – $\text{Al}_2\text{O}_3$. Figures a, b, c, d show the zone of interaction of oxide stiffening elements with the matrix, $\phi$ and $\epsilon$ - there is no interaction between the matrix and the reinforcing filler.

Figure 2. The results of chemical analysis of the composite material VKNA-4U + $\text{HfO}_2$ – $\text{Al}_2\text{O}_3$ oxides,
a - general view of the research field, b - chemical analysis of the matrix (spectrum 51, wt%), c - chemical analysis of the interaction zone (spectrum 50, wt%).
The chemical reaction caused by the interaction of the stiffening element with the matrix leads to a decrease in the strength characteristics of the material [12]. In the process of interaction between the matrix and the filler, brittle metal compounds can be formed [13, 14].

After analyzing the matrix-filler interaction, for further investigation, VKNA-4U + HfO₂ – Y₂O₃ – Al₂O₃ and VKNA-4U + Y₂O₃ – Al₂O₃ alloys were selected, in which the stiffening filler is not dissolved in the matrix alloy.

Also, for all samples it can be seen that nanoscale reinforcing elements are not evenly distributed in the volume of the matrix material. This is due to the low manufacturability of the aggregate fillers, which makes them evenly distributed in the process of mechanical alloying.

To study the physicomechanical characteristics of VKNA-4U + HfO₂ – Y₂O₃ – Al₂O₃ and VKNA4U + Y₂O₃ – Al₂O₃ alloys, samples were made by mechanical alloying followed by compaction by the hybrid spark plasma sintering method [15].

When choosing the optimal content of stiffening components in the cermet powder composition for the matrix of VKNA-4U alloy, the main parameters were the density and impact strength at the room temperature. The choice of the optimal content was carried out in such a way as to ensure the preservation of plasticity at the maximum value of strength.

There were made compact samples of the material containing the reinforcing component 2 wt% and 5 wt%, a sample without a reinforcing component was also made from the matrix alloy. Adding solid stiffening components to the plastic matrix occurs according to the mechanism of plastic deformation inhibition and to a large extent depends on the number and uniform distribution of the stiffening filler by volume of the matrix material (provided there is no interaction between the matrix and the stiffening filler).

The results of studies of density and toughness, bending strength of samples VKNA-4U, VKNA4U + HfO₂ – Y₂O₃ – Al₂O₃, VKNA-4U + Y₂O₃ – Al₂O₃ are presented in table 1. The optimum content of the reinforcing component in the matrix, at which the toughness and flexural strength has a maximum value of 2 wt%, while increasing the content of the stiffening component greatly reduces the ductility of the material.

| Table 1 Results of the study of density and impact strength of samples: pure VKNA-4U, VKNA-4U+HfO₂ – Y₂O₃– Al₂O₃, VKNA-4U +HfO₂ – Al₂O₃ |
|---------------------------------------------------------------|
| Content of reinforcing filler, wt%                          | VKNA-4U | VKNA-4U+HfO₂ – Y₂O₃– Al₂O₃ | VKNA-4U +HfO₂ – Al₂O₃ |
| Density, g/cm³                                   | 7.9     | 7.9                         | 7.9                         | 7.9     | 7.5 |
| Impact strength, kJ/m³                          | 204     | 260                         | 117                         | 149     | 57 |
| Bending strength, MPa                           | 1350    | 1470                        | 1240                        | 1400    | 1170 |

Findings
In this paper, we investigated the influence of stiffening filler for VKNA-4U alloy on it’s mechanical characteristics. The compositions of oxides that do not enter into chemical interaction with the metal matrix were selected as the most perspective for this material. Optimal content was determined for the strengthening component in the matrix, in which the strength characteristics of the material have a
maximum value. An uneven distribution of the reinforcing component during mechanical alloying was established, which requires further refinement and optimization of the process. It is shown that 1-2 wt% of the oxide reinforcing filler increase bending strength by approximately 7.5%. In the future, VKNA-4U powder composition mechanically alloyed with α-Al2O3, Y2O3, HfO2 oxides can be used as a building material for additive manufacturing technologies for power fasteners of gas turbine engines (GTE).

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