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Structure and properties of the nickel aluminide based alloy obtained from a mechanically activated powder mixture by spark plasma sintering

© L.I. Shevtsova[1], M.A. Esikov[1,2], Yu.N. Malyutina[1], A.I. Gavrilov[3], N.Yu. Cherkasova[1], V.N. Malikov[4]

[1] Novosibirsk State Technical University, Novosibirsk, Russia
[2] M.A. Lavrentiev Institute of Hydrodynamics SB RAS, Novosibirsk, Russia
[3] Institute of Solid State Chemistry and Mechanochemistry, SB RAS, Novosibirsk, Russia
[4] Altai State University, Barnaul, Russia
E-mail: edeliya2010@mail.ru

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The paper presents the results of structural studies and evaluation of the strength properties of a domestic Ni_3Al-based alloy grade VKNA-4U obtained from initial components by mechanical activation and subsequent spark plasma sintering. The material was sintered in the temperature range of 1100−1200°C. The highest level of bending strength (1800 MPa) was recorded for the VKNA-4U alloy obtained by heating to 1200°C, a compacting pressure of 40 MPa, and a holding time of 5 minutes. The relative density of the material sintered in this mode is $\sim$ 99%.

Keywords: spark plasma sintering, mechanical activation, nickel aluminide, VKNA-4U alloy, strength properties.

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Nickel aluminide (Ni_3Al) is widely used as a basis for creating structural materials that operate for a long time at high temperatures and in aggressive environments, as a protective oxide layer is formed on its surface [1–3]. By adding various alloying elements to Ni_3Al, a domestic high-temperature structural alloy (HTSA) with the content of ordered γ'-phase (Ni_3Al intermetallic compound) reaching 95% was created. The high content of γ'-phase in the alloy provides thermostable structure and heat resistance up to 1250°C, while the content of less refractory elements leads to lower density compared to nickel super-alloys and higher specific heat resistance [4,5].

In industry, domestic HTSA alloys produced by directional solidification [4–6] are widely used. However, the constant increase in the requirements to the developed materials, as well as the development of powder metallurgy allowed to apply new methods to create and expand the areas of use of alloys with a high complex of properties (including for additive technologies). Improved performance of intermetallic sintered materials can be achieved by grinding intermetallic grains in the consolidated state [7]. Interest in the method of spark plasma sintering (SPS) is due to the possibility of preserving the structural characteristics of the material obtained in powders in the sintered state by reducing the exposure time compared to traditional sintering methods. The structure and properties of nickel aluminide-based alloys produced by spark plasma sintering are under active research [8–10]. In data, the VKNA alloy-4U was first obtained by mixing the initial components, their mechanical activation and synthesis *in situ* by sintering by SPS.

To obtain sintered VKNA-4U alloy, a powder mixture was prepared, the chemical composition of which is shown in Table 1 [6].

Mechanical activation (MA) of the powder mixture was carried out using a high-energy planetary ball mill „AGO-2“ (Russia) in argon with the following processing parameters: the mass ratio of steel balls to powder mixture was 20 : 1; centrifugal acceleration of the balls was equal to 400 m/s^2. Reagent activation was carried out for 3 min 30 s. As a result of this MA mode, dense mechanic composites of lamellar structure were formed (Fig. 1), which were investigated by X-ray diffraction analysis using an ARL X‘TRA diffractometer (USA).

Spark plasma sintering of VKNA-4U mechanic composites was carried out on a LABOX-1575 (Sinter Land, Japan). A graphite mold with the material to be sintered was placed in the working chamber of the unit. Graphite paper 0.2 mm thick was used to protect the mold from the sintered material and to ensure sliding of the punches in the matrix. The mechanical force on the punches was generated by an integrated hydraulic press. Heating was provided by passing a constant pulsating current directly through the mold with a cycle of 40 ms on 12 ms off. Varying parameters were sintering temperature and holding

Table 1. Chemical composition of the powder mixture (in mass%) corresponding to the grade of VKNA-4U alloy (domestic Ni_3Al-based alloy) [6]

Ni	Al	Cr	Mo	W	Ti	Co	C
74.4	8.5	5.0	5.0	2.2	0.9	4.0	0.02

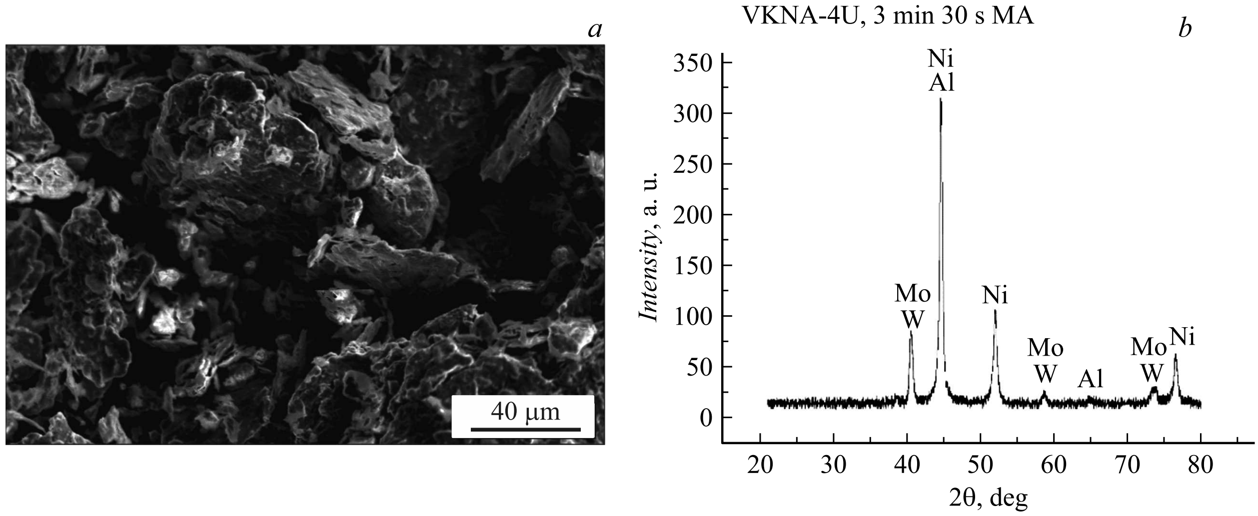

Figure 1. *a* — appearance of the mechanocomposites formed as a result of mechanical activation of the powder mixture corresponding to VKNA-4U alloy; *b* — X-ray diffraction of the mechanocomposite of VKNA-4U composition.

Table 2. Characteristics of SPS sintering modes and properties of obtained compacts from nickel aluminide-based alloy (VKNA-4U)

Number of sample	Grade of alloy	Temperature, °C	Pressure, MPa	Holding times, min	Heating rate, °C/min	Density, g/cm^3	Relative density, % (ref.)	Bending strength, MPa
1		1100		10		7.82	98.85	1215
2	VKNA-4U	1150	40	5	70	7.79	98.48	1370
3		1200				7.85	99.27	1800

time. The range of sintering regimes was determined experimentally, and in view of previous studies [7–9]. As a result of sintering, 40 g mass cylindrical billets with a diameter of 30 mm and a height of 8 mm were obtained. The density of the obtained samples was determined by hydrostatic weighing method. The specimens for the three-point bending tests had the shape of a rectangular parallelepiped with dimensions $3 \times 4 \times 28$ mm. Strength tests were carried out on an Instron 3369 unit. The traverse speed was 0.5 mm/min. Sintering modes and characteristics of the sintered VKNA-4U alloy are given in Table 2.

Table 2 shows that heating to 1200°C increases the density of the sintered VKNA-4U alloy to 99.27%, which in turn has a positive effect on the strength characteristics of the sintered material. Thus, the VKNA-4U alloy obtained by sintering at 1200°C has the highest bending strength, which is 1800 MPa. For the mono-phase intermetallic compound Ni_3Al obtained under similar sintering conditions, the level of flexural strength is 790 MPa. Thus, the introduction of alloying elements makes it possible to increase the bending strength of the powder nickel aluminide based alloy by more than 2 times. The structure of the sintered samples was studied using a Carl Zeiss Merlin scanning-electron microscope equipped with an INCA X-ACT analyzer (Oxfords Instruments) for micro X-ray spectral analysis. It should be noted that the structure of materials obtained by sintering in different modes inherits the lamellar structure of the original mechanic composites (Fig. 2, *a*). Fig. 2 shows the structure of sintered at 1100°C material of VKNA-4U composition as an example. Light-colored regions were recorded in the structure of the material (indicated by arrows in Fig. 2, *a* and *b*). Maps of W and Mo elements in the selected cross-sectional region of the sintered alloy are shown in Fig. 2, *c*. The maps confirm the presence of non-interacting tungsten in the center of the light region and the presence of tungsten and molybdenum around its periphery.

Thus, as a result of spark plasma sintering of the powder composition mechanically activated during 3 min 30 s, which corresponds to the composition of the alloy on the basis of intermetallic compound Ni_3Al of VKNA-4U, samples with density up to 99.27% of the reference density of the specified alloy were obtained. The recommended sintering mode to obtain a dense billet of VKNA-4U alloy with high bending strength (1800 MPa) at room temperature is heating at an average rate up to 70°C/min at a sintering temperature of 1200°C with a holding time of 5 min and an applied uniaxial pressure of 40 MPa.

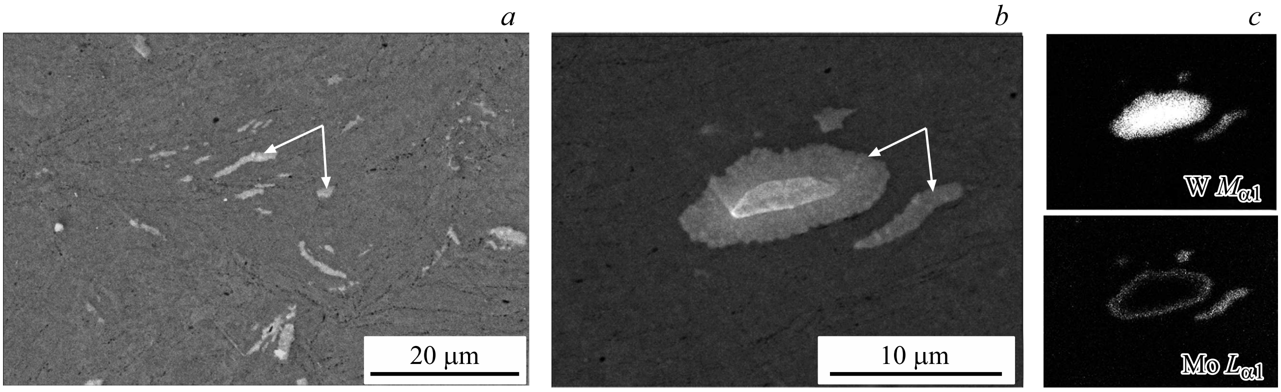

Figure 2. Micrographs of the structure sintered at 1100°C of VKNA-4U alloy (a, b) and maps of the elements fixed in the light region of alloy (c).

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Conflict of interest

The authors declare that they have no conflict of interest.

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