Fabrication of the Ni_3Al-based alloy formed by spark plasma sintering of VKNA powders

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Abstract. The material based on Ni_3Al intermetallic has been obtained from the industrial powder of a VKNA type by the method of spark plasma sintering. Materials sintering was conducted at the temperature of 1100°C, compacting pressure of 20 MPa, and during soaking time equal to 5 minutes. The heating rate of samples amounted to 50 and 200 °C/min. It has been established that the material obtained by sintering at the rate of 50 °C/min possesses a maximum value of density (5.93 g/cm^3) and a maximum level of bending strength (~ 400 MPa).

1. Introduction

A Ni_3Al compound is a basic strengthening phase of heat-resisting nickel alloys used in large quantities when fabricating high-duty parts in chemical industry, power engineering as well as in aircraft industry and rocket production. The content of the Ni_3Al intermetallic in such materials reaches 70 %. The presence of this phase allows using nickel alloys when manufacturing parts operated at elevated temperatures [1]. This is accounted for the combinations of such Ni_3Al properties as heat resistance, oxidation resistance, corrosion resistance at elevated temperatures as well as the high melting temperature (1395 °C) and relatively low density (7.5 g/cm^3) [2-4].

However wide application of the Ni_3Al intermetallic is impeded by a number of peculiarities of nickel aluminides including a low level of plasticity and crack resistance. This peculiarity accounts for intractability of materials by cutting. At room temperature the samples of polycrystalline Ni_3Al intermetallic are brittle [4].

In the past decades the alloys based on nickel aluminide of a VKNA type (Ni_3Al phase content is up to 90 %) developed in the All-Russian Institute of aviation materials (AIAM) have been extensively used. In the technical literature the structure of alloys of the VKNA-type in the as-cast state is described in detail and some information on their mechanical properties are given [2]. However there are not enough data on the structure and properties of such materials obtained by the powder metallurgical technique.

It has been experimentally established that an effective way of obtaining powder compacted materials based on intermetallics is spark plasma sintering (SPS technology) [3]. Owing to a short-term high temperature impact on the powder mixture, an optimal ratio of heating and material deformation modes as well as peculiarities of electric current flow through the sample, SPS technology allows obtaining high strength items with low values of porosity and residual mechanical

stresses. The application of this technology enables preserving a fine-grained structure of the sintered material.

The purpose of this paper is a formation of the material on the basis of nickel aluminide during spark plasma sintering of the powder of the VKNA type at different rates of heating.

2. Materials and methods

The powder of nickel aluminide of the PN75Yu23V (VKNA) grade was used as initial material. The average size of the powder particles was 20 μm. The basic phase of these materials was Ni_3Al intermetallic. The chemical composition of the applied powder is presented in table 1. The material of the VKNA type is applied in industrial production for obtainment of heat and wear resistant coatings, applied to the parts of the machines operating in the hostile environments.

Table 1 – The chemical composition of the nickel aluminide powder of the VKNA grade.

Powder designation	Mass fraction of the elements, % by mass									
	Ni	*Al*	*Cr*	*Co*	*W*	*Ti*	*Mo*	*Ca*	*Fe*	*C*
PN75Yu23V	71.7 -74	19- 22.3	2.93	0.8	1.16	0.32	0.79	0.08	0.15	0.07

The powder was sintered in the spark plasma sintering system 'SPS10-4' produced by Advanced Technology Company (USA). The powder sintering process was implemented as follows: the powder mixture was poured into a current-conducting graphite press mould with the inner diameter of 30 or 40 mm, which was subsequently placed in the vacuum chamber of the installation (10^{-2} Pa).

Sintering was conducted according to the modes presented in table 2. The range of the sintering modes was selected on the basis of literary data and personal studies of the authors of the project.

Table 2 – Modes of spark plasma sintering of the powder of the VKNA type.

Mode number	Powder mixture	Sintering temperature, °C	Compacting pressure, MPa	Soaking time, min.	Heating rate, °C/min	Vacuum, Pa
1	VKNA	1100	50	5	50	10^{-2}
2					100	
3					200	
4			30		100	
5			40			
6			60			

3. Results and discussion

Analysis of the literary data justifies the fact that the process of spark plasma sintering, the structure and properties of the obtained materials is influenced by the following basic parameters of sintering: heating rate, sintering medium, electric current magnitude, compacting pressure, temperature and duration of isothermal soaking [4-7]. Since the most significant difference of SPS technology from hot pressing is the high rate of heating; the problem of its influence on the resulting density of the sintered materials is of great interest. High heating rates allow reducing the sintering process duration and restrict the powder particle growth during the consolidation.

M.S. Boldin's paper [8-10] contains a qualitative analysis of studies on evaluation of the heating rate impact on the structure and properties of the sintered ceramic materials, which specifies the fact that a heating rate increase up to ~ 300 °C/min enables an increase in the density of the finished workpiece. During a subsequent increase of the heating rate, the areas with closed porosity are formed in the material structure, which is accompanied by the density reduction. In this case the density decrease effect is intensified while increasing heating temperature [11]. When studying the nickel

powder sintered at different heating rates (from 90 to 1100 °C/min), the authors of the paper note that high heating rates during sintering lead to heterogeneous compacting of samples and crack formation. On the basis of the analysed literary data VKNA powder heating was realised at the rates of 50, 100 and 200 °C/min. The compacting pressure was varied from 30 to 60 MPa.

Based on the data of studies conducted by the project implementator, the most reasonable sintering temperature of nickel aluminide of the PN85Yu15 grade (an alloy on the Ni_3Al basis) was determined; this temperature allows one to obtain qualitative workpieces possessing the best set of mechanical properties by SPS technology. In this work the sintering temperature remained stable and amounted to 1100 °C.

It is possible to use shorter soaking time compared to customary sintering one for obtaining high-density compacts during SPS. The reduction of soaking time enables reduction of microstructural evolutions as a result of diffusion control of phase transformations. The soaking time for all series of experiments amounted to 5 minutes.

The density of sintered materials was calculated by the method of measurement of sample linear dimensions and by the method of hydrostatic weighing [12]. The obtained averaged values of density of the resulted samples are shown in Table 3.

Table 3 – The influence of compacting pressure and heating rate on the density of the sintered materials.

Mode number	Powder mixture	Compacting pressure, MPa	Heating rate, °C/min	Density, g/cm^3
1	VKNA	50	50	5.93
2			100	5.71
3			200	5.86
4		30	100	5.26
5		40		5.92
6		60		6.23

It is seen from the table that maximum density (6.23 g/cm^3) is reached for the material obtained by VKNA powder sintering at compacting pressure equal to 60 MPa. The reduction of heating rate influences favourably the density of the finished material.

Structural studies of the VKNA sintered materials at different scale levels (with the use of optical metallography, scanning electron microscopy and diffractometry of the X-ray radiation).

Structural studies were conducted under optical and scanning electron microscopes. The scanning microscope of the Carl Zeiss Axio Observer A1m type was used for metallographic studies in the mode of the bright field. For the purpose of structural study under higher magnifications and for X-ray microanalysis a scanning electron microscope of the Carl Zeiss EVO 50 XVP type was used.

The microstructure of the compact sintered by mode №4 is homogeneous throughout the whole volume. Such defects as large pores or cracks were not revealed in the material structure. Formation of the large quantity of micropores in the structure of this material can be explained by the presence of different refractory elements. This testifies the fact that such sintering modes do not provide the formation of high-quality compacts from VKNA powder. The average particle size after sintering by this mode amounts to 30-50 μm. As a result of sintering of the PN85Yu15 type powder under specified modes, a compact with the density of 5.9 g/cm^3, which amounts to 92 % of the Ni_3Al intermetallic density, was obtained.

Figure 1 shows a material structure obtained using the scanning electron microscopy methods. The results of the energy dispersive X-ray microanalysis of the local regions in the sintered material is evidence of the refractory elements allocation (figure 1b).

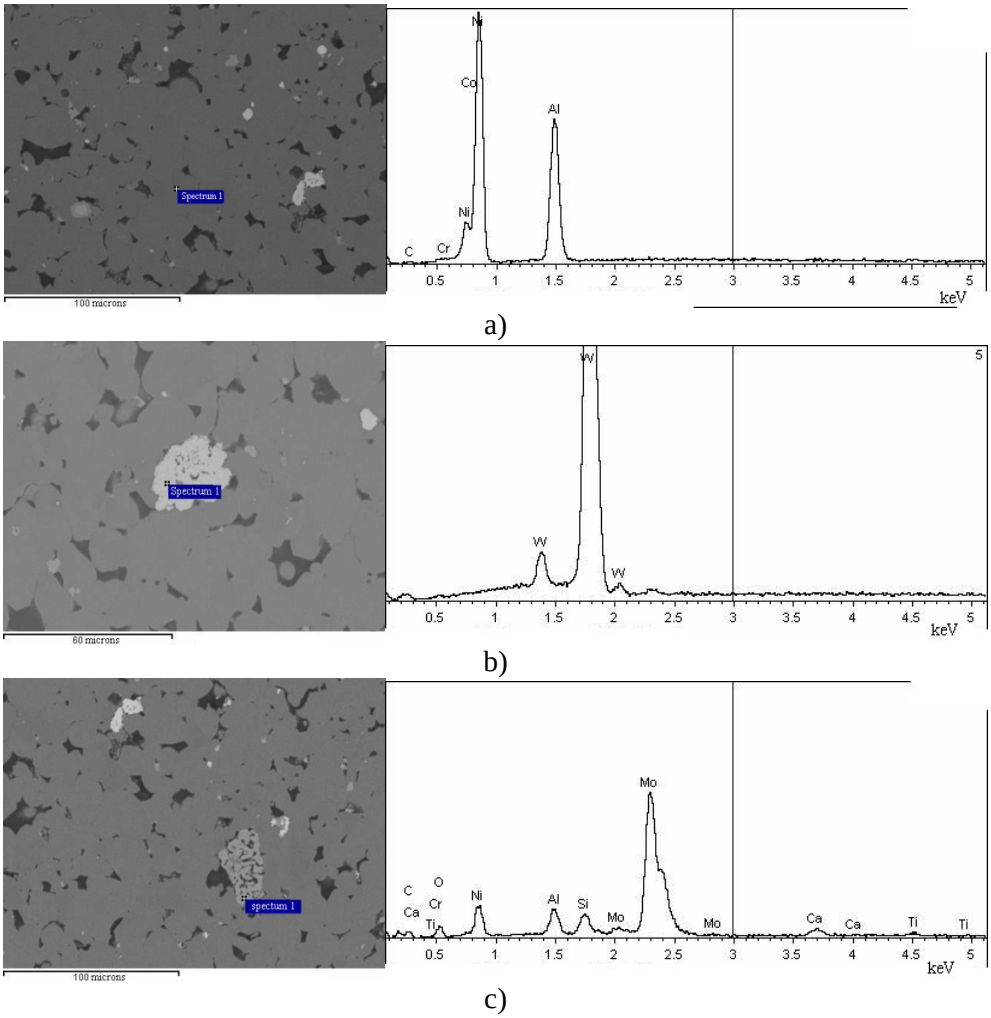


Figure 1. Results of the X-ray microanalysis of the local regions in the material obtained by spark plasma sintering of VKNA powder by mode № 4.

The X-ray phase analysis was conducted using an ARL X'TRA diffractometer. Diffractograms were shot by means of CuKα–radiation with the scanning pitch of 0.02 degree. The accumulation time amounted to 1 s per one point. The X-ray diffraction data analysis allowed determining that the material under study consists of the Ni3Al phase by 95 % with the average size of the coherent-scattering regions equal to 64.5 nm and with microstresses of $1.1 \cdot 10^{-4}$. The remaining 5 % part is an X-ray amorphous phase.

Mechanical tests of the obtained materials comprise determination of the strength properties (durometric studies, strength tests of the materials under conditions of uniaxial tension and three-point bending) and impact toughness.

The hardness indices of most of materials correlate closely with such important mechanical property as the magnitude of ultimate strength. In this case the advantage of durometric studies is simplicity of measurements, applicability of small-size samples, immediacy of experimental data acquisition.

The microhardness of the materials obtained by means of VKNA type powder under different modes was evaluated by means of the microhardness tester, Wolpert Group 402 MVD, intended for Vickers hardness testing. The indentor of the microhardness tester is a tetrahedral diamond pyramid. The indentor pressed on the pyramid with the force of 19.6 N. The measurements were conducted on the crosscut metallographic sections.

Three-point bending tests were conducted to evaluate strength properties. Tests for strength determination under uniaxial tension failed to succeed. This failure is due to increased brittleness of the sintered samples. Bending tests were conducted at room temperature at the installation 'Instron 3369'. The rate of travers travel was 0.5 mm/min. In order to conduct mechanical tests, flat samples with dimensions of 3×4×30 mm were cut out at the electroerosion cutting installation 'Sodick AG 400L'. Testing in the conditions of uniaxial tension of the obtained materials failed to succeed due to increased brittleness of samples.

Impact strength of the sintered samples characterises the energy used for destruction of the workpieces during their dynamic loading. Tests were conducted at room temperature using an impact pendulum-type testing machine 'CAST 9050' (Instron), with the maximum value of impact energy of 25 J. Samples of a rectangular shape with dimensions of 3×4×25 mm (without a notch) were applied for testing. Samples were cut out of the sintered workpieces at the electroerosion cutting installation 'Sodick AG 400L'. In the process of testing the values of fracture energy were fixed.

The results of compacting pressure and heating rate influence on the strength properties are reflected in tables 4 and 5, respectively. The maximum value of the impact strength level is registered on the basis of the material obtained by sintering of VKNA powder under 30 MPa.

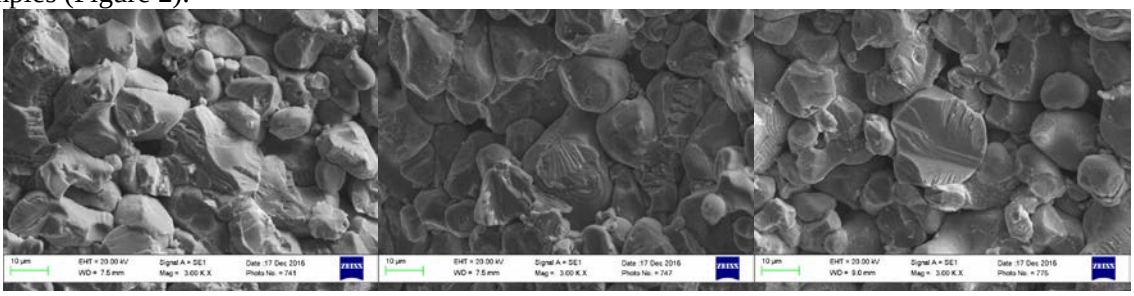
Table 4 – The compacting pressure influence on strength properties of sintered VKNA powder (at heating rate of 100 °C/min).

Powder mixture	Compacting pressure, MPa	Microhardness, HV1	Bending strength, MPa	Impact strength, J/cm^2
VKNA	30	290	220	3.14
	40	480	330	0.8
	50	370	225	0.86
	60	520	490	0.8

Table 5 – The heating rate influence on strength properties of sintered VKNA powder (under compacting pressure of 50 MPa).

Powder mixture	Heating rate, °C/min	Microhardness, HV1	Bending strength, MPa	Impact strength, J/cm^2
VKNA	50	490	390	1
	100	370	225	0.86
	200	415	310	1.05

The results of the fractography studies of the VKNA powder sintered by different modes after bending impact tests indicate a domination of the intercrystalline nature of the fracture in all sintered samples (Figure 2).



a b c

Figure 2. Fractures of samples sintered at different heating rates after bending impact tests: a – 50 °C/min; b – 100 °C/min; c – 200 °C/min.

4. Conclusion

Thus it has been experimentally established that spark plasma sintering is an effective method of obtaining low defective compacts from the materials based on nickel aluminide with the enhanced set of mechanical properties. This technology allows preserving a fine-grained structure of the sintered material. Optimization of the spark plasma sintering modes was conducted using literary data and experimental research. It has been revealed that such sintering parameters as compacting pressure and heating rate play an important part in formation of the structure and properties of the sintered material. It has been established that an increase in the compacting pressure enables an increase in the relative density of the material and strength properties of sintered VKNA powder. The maximum value of bending strength (490 MPa) is registered in the material obtained by sintering at the pressure of 60 MPa. The reduction of the heating rate also influences favourably the density and strength properties of the sintered material.

Based on the obtained experimental data it has been established that it is expedient to conduct VKNA powder sintering in the following mode: the heating temperature of the sample should be 1100 °C; the average heating rate is 100 °C/min; compacting pressure and soaking time under pressure at sintering temperature should be equal to 60 MPa and 5 minutes, respectively.

5. Acknowledgements

The work has been performed with financial support of the Russian Foundation for Basic Research, grant № 15-33-50845 (mol_nr) and Russian President Grant (SP-1179.2015.1).

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