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Application of HP-HT methodin the manufacture of NiAl phase

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ABSTRACT

Purpose: The aim of the study was to determine the effect of variable temperature and constant pressure to consolidate the powder mixture of nickel and aluminum in an amount to provide receipt of NiAl intermetallic phase. In particular, the influence of high pressure - high temperature (HP-HT) method to change the structure of sintered density, porosity, Young's modulus and Vickers hardness.
Design/methodology/approach: Using the parameters of the sintering pressure of 7±0.2 GPa and a temperature of 1300, 1400 and 1500°C formation during 60 s, using a HP-HT Bridgman, sintered intermetallic phase NiAl. Sinters were obtained X-ray qualitative analysis, as well as the physical properties of the selected set: the density and Young's modulus. Strength properties of hardness were determined in an attempt to Vickers. Microstructural observations were performed on an optical and scanning microscope, EDS analysis was performed also received NiAl phases.
Findings: Manufactured by HP-HT phase NiAl characterized by compact design with relatively low porosity. Microscopic observations and EDS analysis revealed the existence of a complex multi-phase structure, which also resulted from microhardness tests. It was found that the optimum properties have produced NiAl phase at the sintering temperature of about 1300°C.
Research limitations/implications: Experimental confirmation of the possibility of producing sintered metal powder HP-HT method implies the desirability of extending research in the direction of increased property including through the addition of other elements or ceramic compounds, heat treatment processes, and also via a change in pressure - which is one of the main parameters of the sintering process.
Practical implications: Sintered NiAl phases can be used as a matrix composites reinforced with ceramic particles. Composite based on intermetallic phase NiAl as a result of strengthening the ceramic particles should have correspondingly high strength properties at elevated temperatures, as well as resistance to oxidation and abrasion.
Originality/value: Used for the manufacture of NiAl intermetallic phase sintering technology under high pressures (HP) and the temperatures (HT), which then parameters were well-chosen to get optimum usable properties (mechanical and physico-chemical properties). In addition, the studied process characterized the short time of consolidation of powders as well as the possibility to obtainment of assumed chemical composition.
Keywords: Production of sintered Ni-Al; HP-HT method Bridgman type; Two-phase alloys NiAl/Ni_3Al

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1. Introduction

For many years, the intermetallic phases Ni-Al system (NiAl, Ni_3Al) are the subject of intense study, as they are seen as a potential structural material suitable for work in a wide range of temperatures.

The β-NiAl (B2 structure) intermetallic is known to possess useful properties such as low density (5.91 g cm^{-3}), a high melting point (1638°C), good high-temperature oxidation and corrosion resistance, as well as good thermal conductivity (75W m^{-1} K^{-1}). However, the lack of room temperature ductility and the low high-temperature strength have hindered commercial application of this alloy. Therefore, in order to accommodate phase NiAl for construction applications, it has been intensive research focused on improving the mechanical properties [1,2].

Many researchers have found that microstructural modifications from β-NiAl including the ductile γ` phase result in remarkable enhancements of hot workability and room temperature ductility [3,4].

To obtain the fine distribution of the γ' phase in the β matrix, many researchers have used the martensitic transformation of NiAl [5]. These results, along with the fact that the β phase is capable of undergoing thermoelastic transformation from β to β' ($L1_0$ structure) in certain composition ranges [6], open up a wider range of possibilities for microstructural control and practical application.

Intermetallic phases have been prepared by a variety of methods including casting, mechanical alloying, gas atomization, sintering, combustion synthesis, etc. [7-11].

The main objective of this study was manufacture a high pressure consolidation technique nickel and aluminum powders, sintered NiAl phases, characterized by minimal porosity, fine-grained structure and mechanical properties of isotropy. The study was used Bridgman device that provides smooth control of temperature (up to 2500°C) and pressure (4-8.5 GPa) [12-15]. Structural analyzes were carried out on an optical microscope and SEM, and mechanical properties were evaluated based on the Vickers hardness and Young's modulus.

2. Experimental procedure

The aim of the study was to manufacture using HP-HT (high pressure-high temperature) sintered NiAl alloys and evaluation of their structure and selected properties. The study used Ni79Al21 alloy powder (Goodfellow company), which have grain 45-150 μm. The powders were in the initial state X-ray phase identification. Sintering powders carried out using a pressure sintering apparatus HP-HT Bridgman type.

The powders were placed in a graphite heater with an internal diameter of 15 mm, and then a special batch reaction, providing electrical contact and pseudo isostatic sintering conditions. In order to determine the actual parameter scale pressure sintering was carried out for a given temperature and the reaction cartridge. The pre-formed mixture was placed in a graphite heaters which are subject to sintering at a pressure of 7±0.2 GPa, for 60 seconds at three different temperatures close to melting point: 1300, 1400 and 1500ºC. Diagram of equipment and method of sintering are shown in Figure 1.

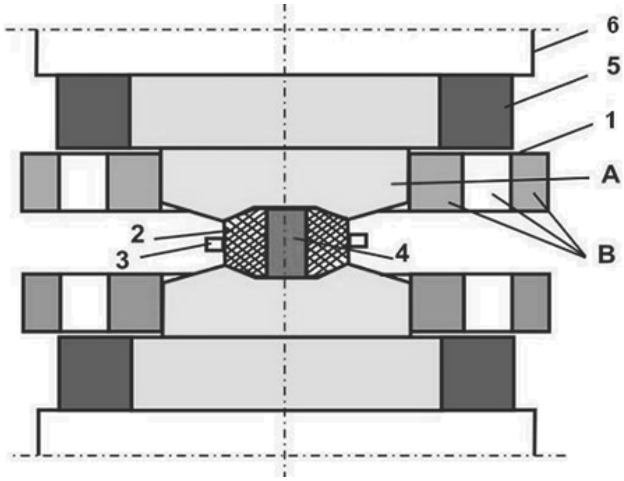

Fig. 1. Scheme of Bridgman-type, toroidal HP-HT apparatus: 1 - anvil (A - central part made of sintered carbides, B - supporting steel rings), 2- pyrophyllite container, 3 - pyrophyllite gasket, 4 - material for sintering, 5 - punch, 6 - supporting plate [12]

Produced in the sintering process disks with dimensions: diameter 15 mm, height of 5 mm were machined. Control alloy phase composition was evaluated by X-ray phase identification (qualitative analysis phase).

Measurements were also carried out of selected physical properties: Young's modulus, density, using the method: ultrasonic, hydrostatic, respectively, and the Vickers hardness measured using microtester FM-7 with 0.98 N load. Young's modulus was determined using ultrasonic flaw detector Panametrics Epoch III. Measurement uncertainty in determining the density was 0.02 g m^{-3}.

In addition, observations were obtained structural alloys suitably prepared surface with an optical microscope Olympus GX-51 (observation in polarized light and Nomarski contrast), and scanning electron microscope JEOL JSM 6460 LV with the possibility of EDS analysis of the chemical composition. Samples were etched with chemical reagent of the following composition: 45% CH_3COOH, 35% HNO_3, 10% HCl, 10% H_3PO_4. These studies made it possible to assess the homogeneity of the microstructure of sintered alloys.

3. Results and discussion

Used for the commercial production of sintered powders (Ni79Al21) was subjected to X-ray identification phase, the results of which are shown in Figure 2.

X-ray analysis revealed the existence of two-phase structure ($NiAl/Ni_3Al$) test powder.

A similar analysis for the samples subjected to the high pressure sintering at different temperatures did not show changes in the two-phase structure.

Qualitative phase analysis of Figure 3, the material sintered by HP-HT: Sample 1 - sintering temperature of 1300°C, Sample 2 - sintering temperature of 1400°C, Sample 3 - sintering temperature of 1500°C.

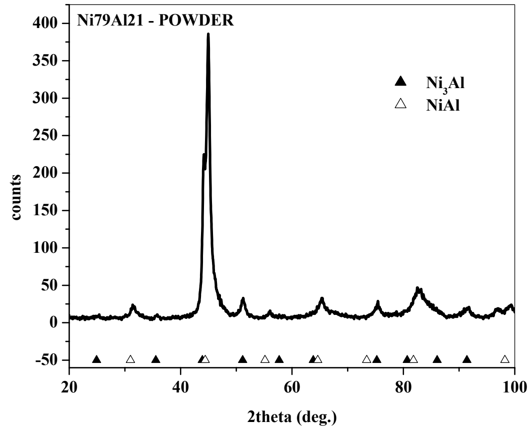

Fig. 2. Qualitative phase analysis of the powder in the initial state Ni79Al21

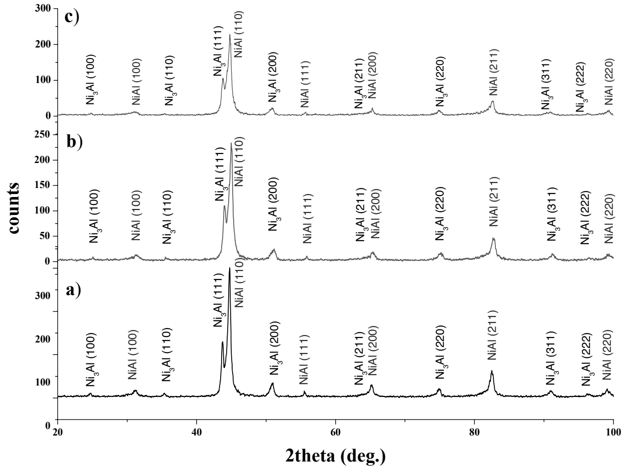

Fig. 3. Qualitative phase analysis of the material sintered by HP-HT: a) Sample 1 - sintering temperature of 1300°C, b) Sample 2 - sintering temperature of 1400°C, c) Sample 3 - sintering temperature of 1500°C

Sintering parameters and physical properties of the set of samples are shown in Table 1 and the changes of the Young's modulus in Figure 4.

The density of the samples produced an average of 6.6 g cm^{-3}, a value is convergent with the theoretical density of the alloy, and corresponds to the theoretical density of about 0.89 and 1.12 for Ni_3Al and NiAl phase, respectively. No significant differences in density depending on the sintering temperature.

Young's modulus of samples determined ultrasonic method, showed lower values (average of 140 GPa), which is about 0.85 and 0.75 for Ni_3Al and NiAl, respectively. There was a slight difference in the Young's modulus according to the sintering temperature. The samples sintered at high temperature (1500°C) showed the lowest value of the Young's modulus.

Table 1 presents the results of measurements of the Vickers hardness, and Figure 5 shows the change in Vickers hardness, depending on the sintering temperature of the samples.

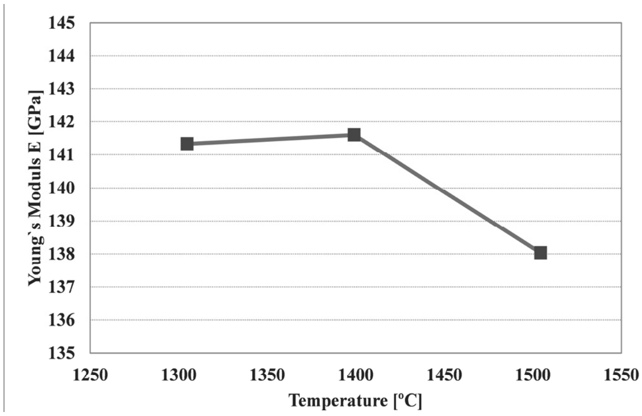

Fig. 4. The change in Young's modulus depending on the sintering temperature by HP-HT for Ni-Al

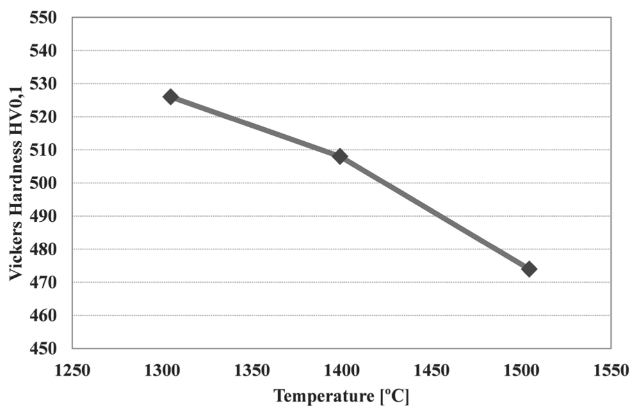

Fig. 5. The dependence of microhardness Vickers HV0.1 from sintering temperature of samples

Table 1.
Sintering parameters and some physical properties

Designation	Pressure [GPa]	Temperature [°C]	Sintering time, [s]	density [g/cm^3]	Poisson`s ratio	Young`s modułus E, [GPa]	Microhardness HV0.1
Sample 1	7.5	1305	60	6.59	0.36	141	526
Sample 2		1399		6.64	0.36	142	508
Sample 3		1504		6.61	0.36	138	474

Analysis of the results of measurement of the Vickers hardness (HV0.1) revealed significant differences in the different hardness values of the same sample. The diverse composition of the phase structure significantly affected the spread of measurements, reflecting the contribution of each phase at the point of measurement.

Average Vickers hardness values were the highest for the samples sintered at the lowest temperature (1300°C). Vickers hardness measurements were made in a single grain, similar measurements made on the grain boundary is characterized by significantly higher hardness values, for example for sample 1, about 754 HV0.1.

Images structures taken on an optical microscope sample 2 contains indigestible in Figure 6, and after digestion in Figure 7. The observed microstructure without voids, compact design (minimal porosity) and visible despite the diverse micro-construction phase.

Also made observations of the microstructure of the scanning electron microscope, images for each sample is shown in Figures 8-10.

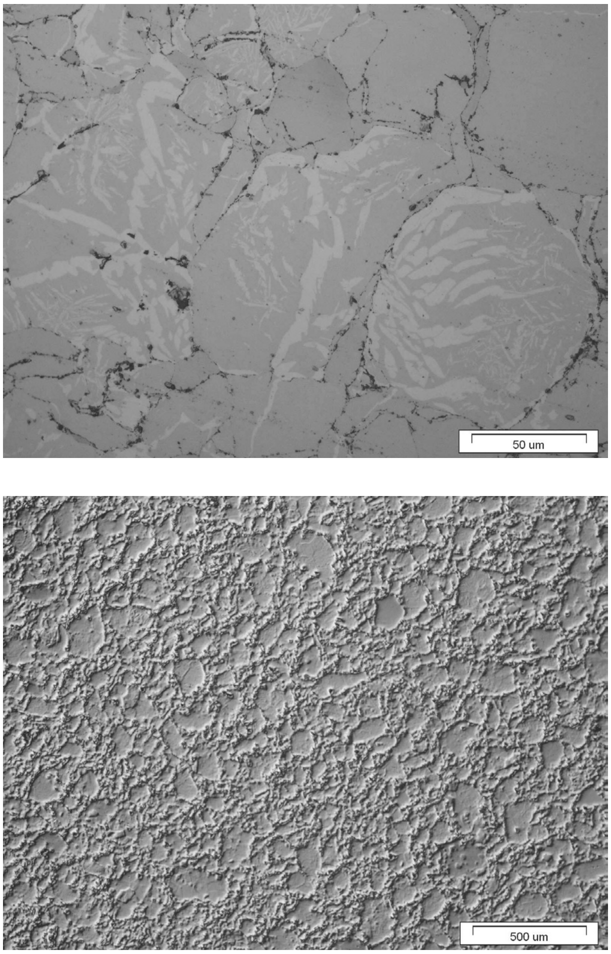

Fig. 6. Sintered microstructure HP-HT, Ni-Al alloy (Sample 2), an optical microscope, before etching

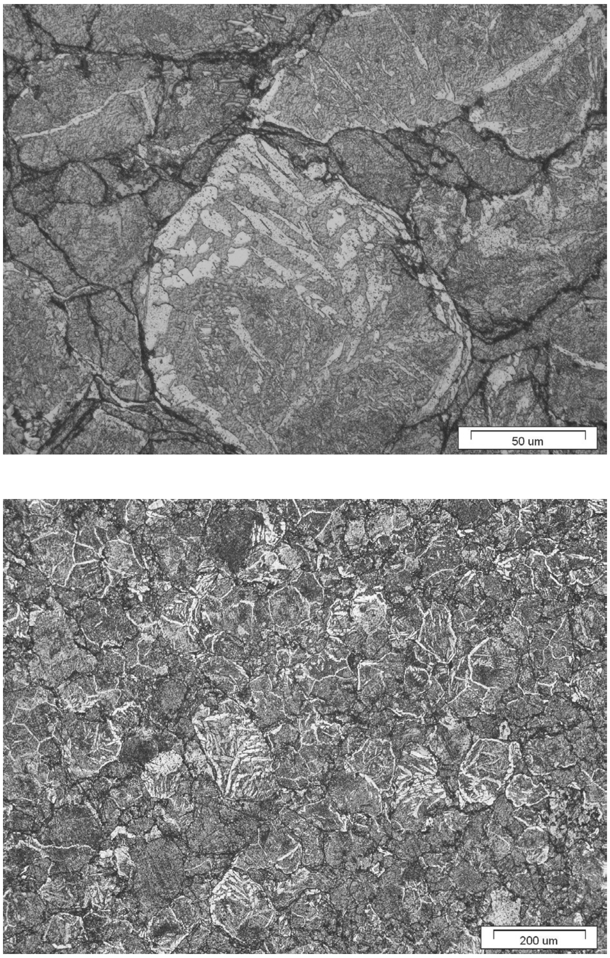

Fig. 7. Microstructure of sintered HP-HT method NiAl alloy (Sample 2), an optical microscope, after etching

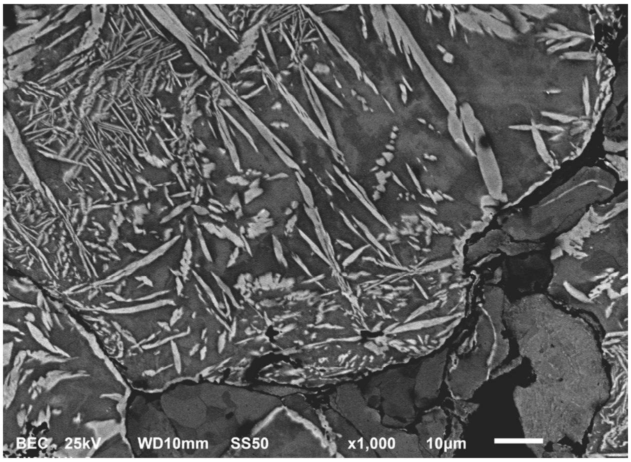

Fig. 8. Microstructure of the sintered HP-HT, Ni-Al alloy (Sample 1), SEM, 1000x

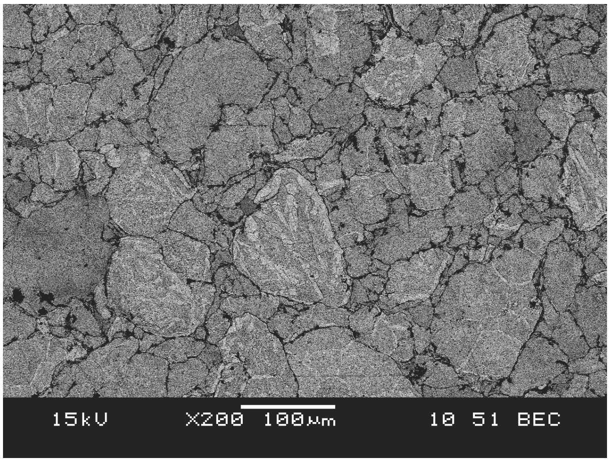

Fig. 9. Microstructure of the sintered HP-HT, Ni-Al alloy (Sample 2), SEM, 200x

The results of EDS analysis of specific points of the sample 1 is shown in Figure 11.

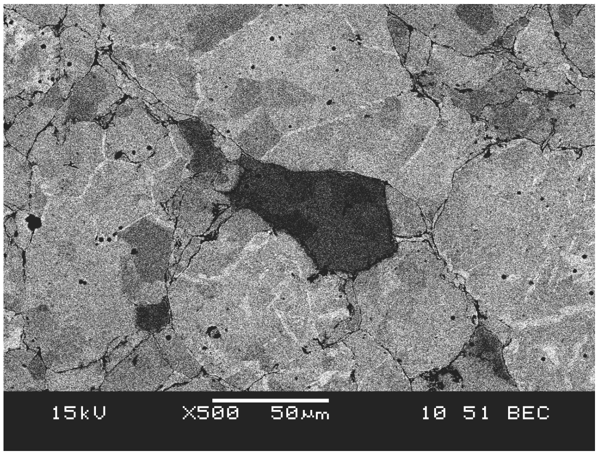

Fig. 10. Microstructure of the sintered HP-HT, Ni-Al alloy (Sample 3), SEM, 500x

Microstructural observations in conjunction with the analysis of the phase structure confirmed the existence of a complex construction phase sintering, with a relatively even distribution of surface elements (Fig. 12).

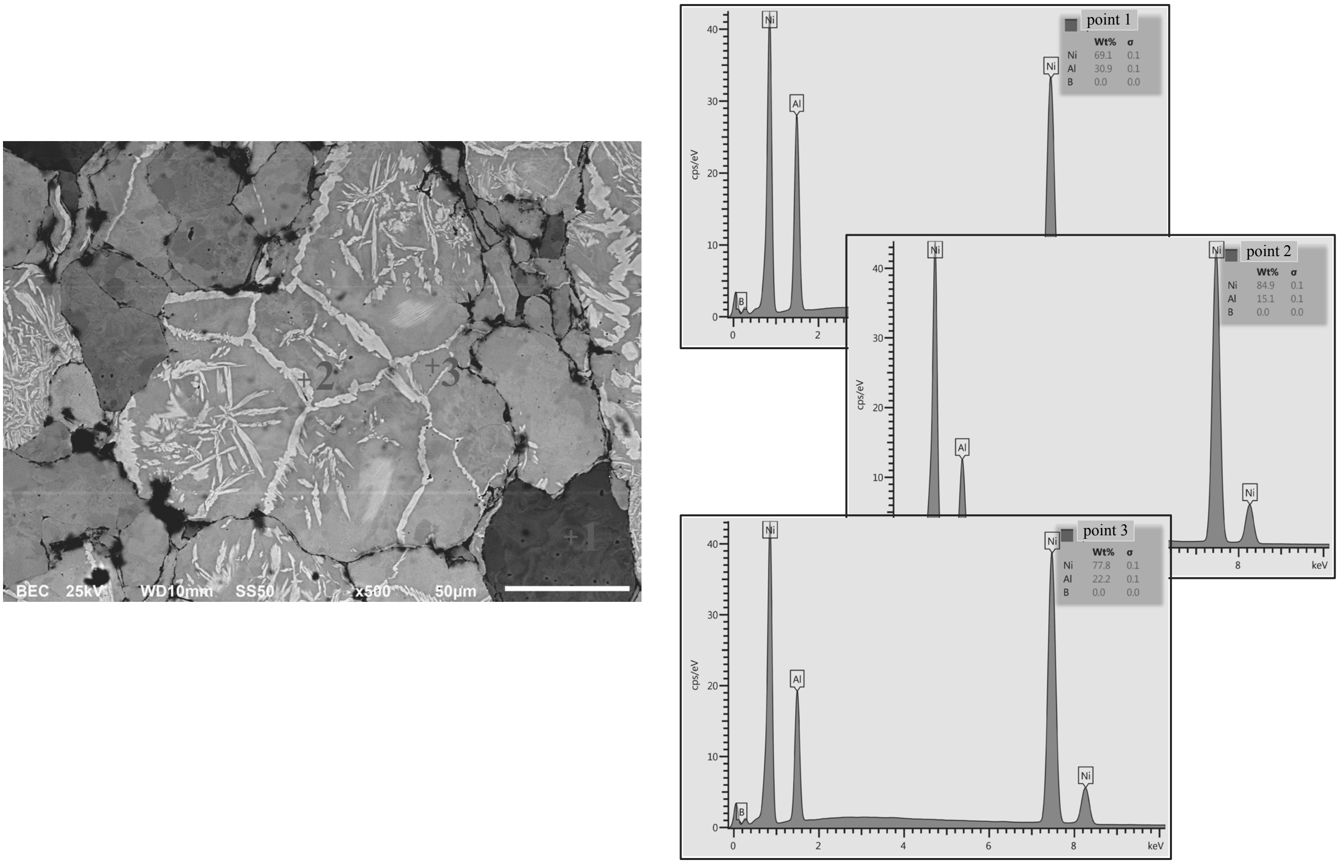

Fig. 11. EDS analysis point, NiAl alloy (sample 1), SEM

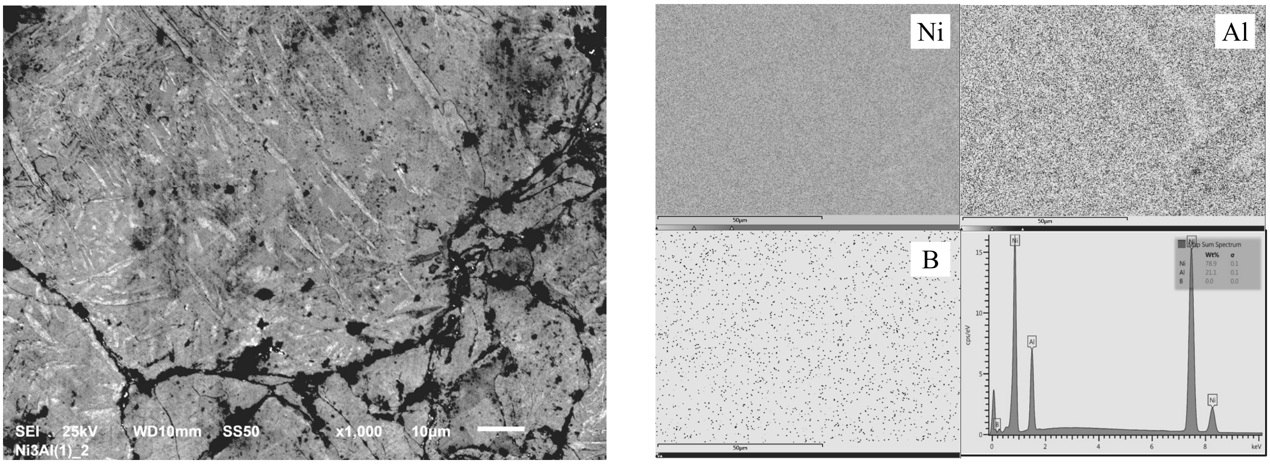

Fig. 12. Distribution of surface elements, NiAl alloy (Sample 1), SEM

4. Conclusions

1. Experimental results have shown the possibility of producing sintered powder commercial Ni79Al21, HP-HT method Bridgman type, applied pressure in a row 7 GPa and the temperature of the process about 1300, 1400 and 1500°C.
2. Alloys so produced are characterized by the structure of the compact with a minimum porosity. X-ray phase analysis revealed the existence of a two-phase structure of the sintered $NiAl/Ni_3Al$.
3. Sintered characteristic values: density - 6.6 g cm^{-3}, Young's modulus - 140 GPa. Optimal size occurred in the sinter produced at 1300°C: density - 6.59 g cm^{-3}, Young's modulus - 141 GPa. The sinter produced in these conditions of pressure and temperature of the highest average value of microhardness 526 HV0.1.
4. Demonstrate the ability to produce sintered powder Ni79Al21, HT-HP method justifies continued research achievements in terms of improved properties by changing the parameters of the process, the addition of other elements or compounds, or heat treatment.
5. The tested alloy can be used as the matrix of the composite reinforced with ceramic particles. Composites of this type so far produced by the process of casting or long sintering.

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