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Boron-modified Ni_3Al intermetallic compound formed by spark plasma sintering of mechanically activated Ni and Al powders

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Abstract. A Ni_3Al intermetallic compound was obtained by spark plasma sintering of mechanically activated Ni and Al powders in atomic ratio 3:1 respectively. Samples with boron addition of 0.1 and 0.2 % (wt.) and samples without boron were obtained. The maximum value of the relative density (~ 99 %) has been obtained for the material by sintering of mechanically activated mixture powders modified with 0.1 % of boron. No differences have been found between the structure of boron-modified Ni_3Al and Ni_3Al without boron addition. The maximum level of bending strength (2200 MPa) has been achieved for Ni_3Al with 0.1 % (wt.) of boron. This value is almost 3 times the bending strength of the sample of Ni_3Al sintered without boron addition.

1. Introduction

Development of the materials with enhanced physical, chemical and mechanical properties is one of the topical issues of advanced materials science. Particular attention is paid to the investigation of nickel aluminides due to their high mechanical properties, high corrosion resistance and oxidization resistance at high temperature [1-5]. Among all aluminides Ni_3Al has anomalous temperature dependence of the yield strength in a certain temperature range. This phenomenon is advantageous from the point of application of the intermetallic compounds as a structural material in aircraft and rocket industry, chemical engineering and power engineering [1-3].

However, the widespread industrial application of nickel aluminide is constrained by a number of features including the low level of ductility and fracture toughness. This fact explains the poor machinability of materials. Polycrystalline Ni_3Al is brittle at room temperature [4].

In recent years, heightened interest has been aroused in the obtaining of materials based on intermetallic compounds produced by mechanical activation of powder mixtures and subsequent spark plasma sintering [3-9]. Mechanocomposites with significant dispersion of reagents are formed as a result of plastic deformation during mechanical activation of powder mixture in a planetary ball mill. Mechanocomposites are characterized by an increasing contact area, and a large number of nonequilibrium defects and internal stresses [8]. Due to short duration of high temperature exposure, an optimal ratio of heating and deformation modes, and features of current flow through the powder mixture, SPS-technology allows producing high-strength materials with the low level of porosity and low residual stresses. Application of this technology promotes the formation of a fine grain structure. However, intermetallic compounds obtained using the technology described above still has low ductility [6, 7, 9-12].

It is well known, that one way to improve the ductility of the intermetallic Ni_3Al while increasing its mechanical properties is microalloying. The analysis of published data shows that the most effective alloying element for Ni_3Al is boron. The positive effect of boron on the plasticity of the intermetallic is related to its segregation to the grain boundaries. However, this method works only as long as the content of aluminum in the alloy does not exceed 25 % (at.). In addition, the grain size should be sufficiently fine [13-15].

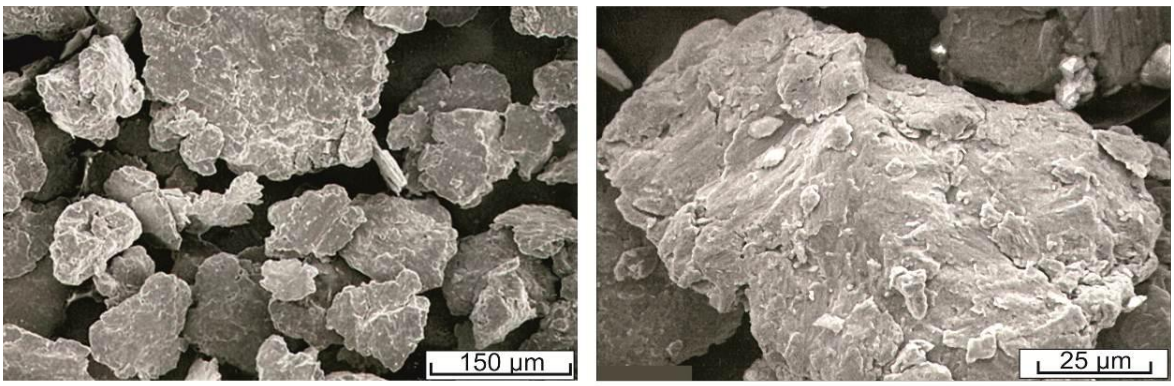
In paper [15] the authors investigated the dependence of boron concentration on the mechanical properties. Samples with 0.2 % (wt.) of boron demonstrated the highest level of mechanical properties. Further increasing of boron concentration led to the embrittlement of Ni_3Al because of the borides formation. Thus, in this paper the effect of boron microadditions on the structure and mechanical properties of intermetallic compound Ni_3Al formed by SPS-sintering of mechanically activated powders of nickel and aluminum were investigated.

2. Materials and methods

Carbonyl nickel powder PNK UT3 (99.85 % Ni) and aluminum powder PA-4 (98 % Al) with the average particle size of 10 μm and 60 μm, respectively, were used as initial materials. The ratio of nickel and aluminum powders was 3:1, which provided the formation of the $Ni_{75}Al_{25}$ % (at.) compound. Amorphous boron was chosen as a modifying element. The amount of added boron was 0.1 and 0.2 % (wt.).

Preliminary activation of the reaction mixtures was carried out in two steel drums of the planetary ball mill ‘AGO-2’. Each drum was filled with 10 g of the powder mixture. The weight ratio of the balls and the powder mixture was 20:1. In order to prevent oxidation of the materials the drums were filled with argon, the pressure of the gas was 0.3 MPa. Centrifugal acceleration of balls reached $400\ m \cdot s^{-2}$.

Experimental results and literature data revealed the optimum time (3 min) for mechanical activation which ensures the formation of single-phase intermetallic compound Ni_3Al during the subsequent sintering of this mixture [8, 10, 11]. Mechanocomposites consisting of flake shaped nickel and aluminum particles were prepared after three minutes of mechanical activation of powder mixtures (Figure 1).



a b

Figure 1. Mechanocomposites based on nickel and aluminum formed after 3 minutes of mechanical activation.

SPS-sintering of mechanocomposites was carried out using the SPS Labox-1575 equipment in a graphite mold with the inside diameter of 30 mm. Heating speed of the powder mixture was 100 °C/min, pressing pressure was 40 MPa. The samples were sintered at 1100 °C during 5 min. The density of the obtained materials was calculated by determining their mass and linear dimensions.

Structural studies of sintered materials were performed using scanning electron microscopy. The phase composition of the samples was determined using an X-ray diffractometer 'ARL X`TRA' with the copper tube X-ray source. Diffraction patterns were recorded with a step of Δ2θ and the dwell time of 5 sec.

The value of bending strength was used as the main indicator of the strength properties. Samples for the bending test (a rectangular shape with dimensions of 3×4×28 mm) were cut using an electrical discharge machine 'Sodick'. Mechanical three-point bending tests were performed at room temperature using the 'Instron 3369' machine. Crosshead speed was 0.5 mm/min.

3. Results and discussion

Radiographs of materials obtained by sintering of mechanically activated Ni and Al powder mixtures without boron addition and with small amount of boron are shown in Figure 2. The X-ray analysis of sintered materials free from boron indicates the formation of only a Ni_3Al phase. The results confirm that mechanical activation and subsequent sintering according to the present modes provide the formation of single-phase nickel aluminide. Radiographs of sintered materials modified with boron also showed only a peak of nickel aluminide. Reflexes corresponding to boron compounds were not detected. This is due to the amorphous structure of boron and its small amount (up to 0.2 % (wt.)).

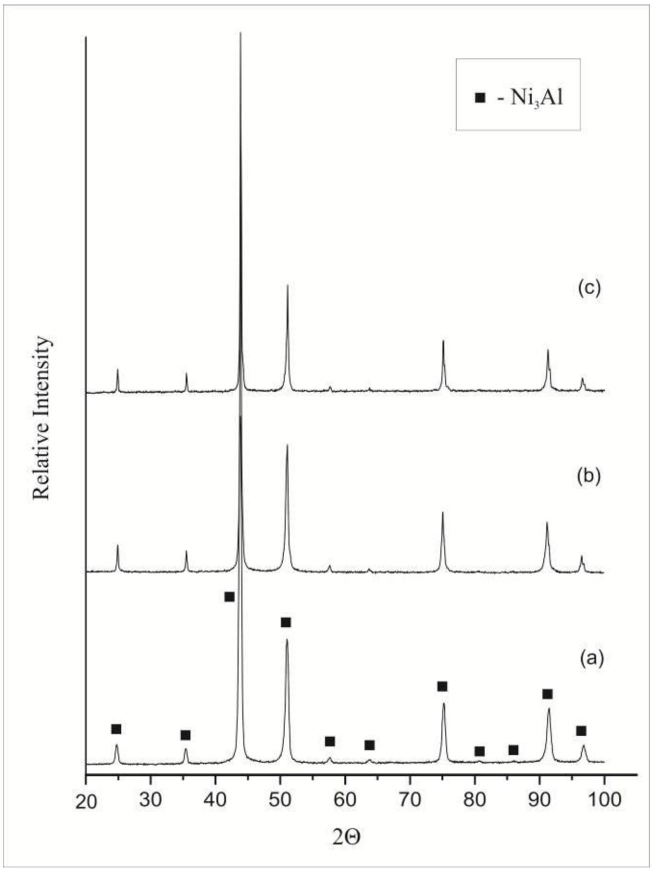


Figure 2. The XRD patterns of samples obtained by sintering of the mechanically activated powder mixture of nickel and aluminum with no boron addition (a); with boron addition of 0.1 % (wt.) with boron addition of 0.2 % (wt.)

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The effect of boron addition on density of the sintered compounds is represented in Table 1.

Table 1. The effect of boron addition on density and relative density of Ni_3Al obtained using an SPS-technology.

Boron content, % (wt.)	Density of sintered samples, g/cm^3	Relative density, %
–	7.29	97.2
0.1	7.41	98.8
0.2	7.39	98.6

The results in the table indicate that boron has a positive effect on the density of sintered intermetallic Ni_3Al. It should be noted that increasing the boron concentration from 0.1 to 0.2 % (wt.) did not significantly affect the density of the sintered sample. The maximum value of relative density (98.8 %) was observed in the sample obtained by SPS-sintering of mechanically activated samples with 0.1 % (wt.) of boron addition. This fact allows expecting that the resulting material will have an increased complex of mechanical properties as compared to Ni_3Al intermetallic compound sintered without modifying elements.

The structure of the boron-modified sintered material was revealed using a scanning electron microscope. No significantly structural differences from the boron-free materials were found. As an example, the structure of the material obtained by the sintering powder mixture with 0.1% addition of boron is illustrated in Figure 3.

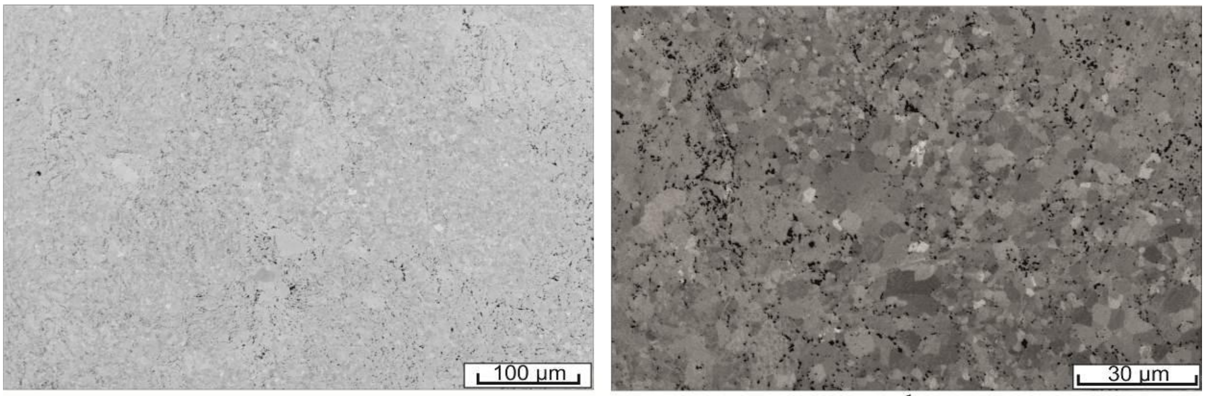

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Figure 3. The structure of the samples obtained by SPS-sintering of the mechanically activated powder mixture of nickel and aluminum with 0.2 % (wt.) addition of boron.

The influence of boron addition on strength characteristics of aluminide nickel produced by the SPS-sintering method is shown in Table 2.

Table 2. The influence of boron on the bending strength of sintered Ni_3Al

Composition of the material	Bending strength, MPa
Ni_3Al	790
Ni_3Al + 0.1 B % (wt.)	2200
Ni_3Al + 0.2 B % (wt.)	1720

The value of bending strength of the Ni_3Al intermetallic compound obtained by the spark plasma sintering technology of nickel and aluminum mechanically activated powders was ~ 800 MPa. A typical fracture behavior of the material is shown in Figure 4. The fracture pattern revealed brittle fracture of the sample.

As it has been noted above, the modification of the nickel and aluminum powder mixture with 0.1 ... 0.2 % (wt.) of boron improves the density of sintered Ni_3Al intermetallic. The maximum level of strength properties was obtained when testing the material containing 0.1 % (wt.) of boron. The bending strength (2200 MPa) of this sample is almost 3 times the bending strength of nickel aluminide sintered without boron addition. The fracture surface of the sample sintered with 0.1 % (wt.) of boron contains pits that are typical of ductile fracture. Areas with signs of brittle fracture were considerably reduced (Figure 4b).

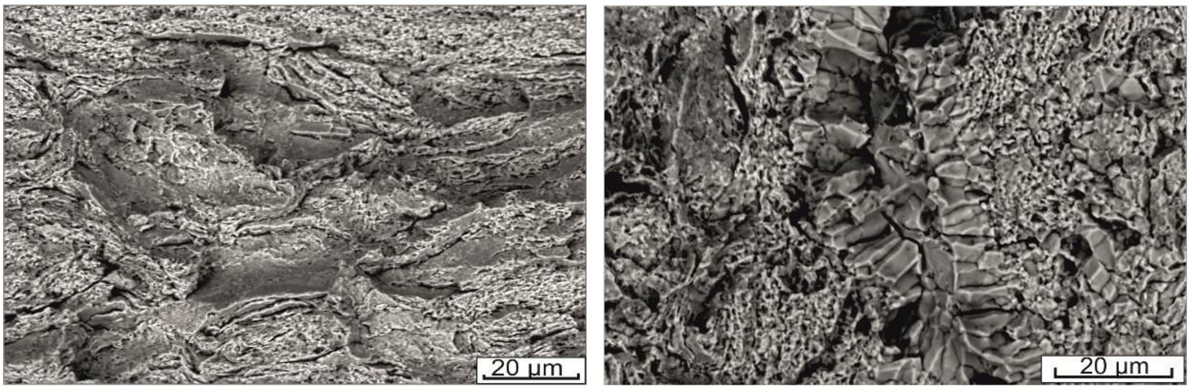

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Figure 4. Fracture surface of the samples of Ni_3Al, obtained by SPS-sintering of the mechanically activated powder mixture without boron addition (a), and with addition of 0.1 % (wt.) of boron (b)

4. Conclusion

Applying the processes of mechanical activation of powder mixture free from boron, and with 0.1 and 0.2 % (wt.) of boron addition and subsequent spark plasma sintering allows obtaining a Ni_3Al intermetallic compound.

Addition of small amount of boron to the initial mixture of nickel and aluminum improves the density of the sintered Ni_3Al intermetallic compound. The maximum value of the relative density (~ 99 %) belongs to the material obtained by sintering of the mechanically activated powder mixture with addition of 0.1 % of boron. The only peaks corresponding to Ni_3Al were revealed in XRD patterns of materials sintered with boron addition. Reflexes corresponding to boron compounds were not found due to their small amount and amorphous structure.

The analysis of the obtained data indicates that addition of small amount of boron to the powder mixture of nickel and aluminum had a positive effect on the mechanical properties of the intermetallic compound obtained by the SPS-method. The samples containing 0.1 % (wt.) of boron showed the highest level of bending strength (2200 MPa). This value is almost 3 times the bending strength of nickel aluminide sintered without boron addition. The fracture surface contains pits that are typical of ductile fracture. Areas with signs of brittle fracture were considerably reduced.

5. Acknowledgements

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