ARCHIVES OF METALLURGY AND MATERIALS
Volume 51 2006 Issue 1

M. ROZMUS*, M. BLICHARSKI*, S. DYMEK*

EFFECT OF TITANIUM ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MECHANICALLY ALLOYED Nb-Ti-Al ALLOYS

WPŁYW TYTANU NA MIKROSTRUKTURĘ I WŁASNOŚCI MECHANICZNE STOPÓW Nb-Ti-Al WYTWARZANYCH MECHANICZNĄ SYNTEZĄ

The goal of this work was to analyse the influence of titanium on microstructure and mechanical properties of mechanically alloyed Nb-Ti-Al alloys. Two alloys composition: Nb15Ti15Al and Nb22Ti15Al (all in at. %) were chosen. The X-ray diffraction of both milling alloys showed that 100 h of mechanical alloying produces the niobium solid solution phase (Nb_{ss}). Powders after 100 h of mechanical alloying subjected annealing at 1000°C were composed of Nb_{ss} and the Nb_3Al intermetallic phase. The alloy with 15% Ti after consolidation was composed mainly of three phases: Nb_{ss}, Nb_3Al and dispersoid, while the alloy with 22% Ti contained additionally very brittle and unfavourable Nb_2Al intermetallic phase. The presence of the detrimental Nb_2Al phase lowers considerably the hardness of the produced alloy. Relatively low fracture toughness of both alloys suggests that the incorporation of a ductile phase (Nb_{ss}) into microstructure is not sufficient for improving the ductility of the synthesized alloys.

Keywords: Niobium aluminides, mechanical alloying

Celem pracy była ocena wpływu zawartości tytanu na mikrostrukturę i własności mechaniczne stopów z układu Nb-Ti-Al, wytwarzanych mechaniczną syntezą. Badania przeprowadzono na dwóch stopach z układu Nb-Ti-Al, różniących się zawartością tytanu. Zawartość Ti w jednym stopie wynosiła 15%, a w drugim 22%. Rentgenowska analiza fazowa wykazała, że oba stopy po 100 h mielenia składały się z roztworu stałego niobu Nb_{ss}. W proszku mechanicznie stopowanym przez 100 h, a następnie wyżarzonym w temperaturze 1000°C przez 5 godzin występowała również faza międzymetaliczna Nb_3Al. Stop zawierający 15% Ti, po konsolidacji składał się z roztworu Nb_{ss}, fazy Nb_3Al oraz dyspersoidu tlenkowego, podczas gdy w stopie o większej zawartości tytanu dodatkowo występowała faza Nb_2Al. Obecność fazy Nb_2Al o bardzo dużej kruchości spowodowała, że twardość stopu z 22% Ti jest mniejsza niż stopu z 15% Ti. Stosunkowo niska odporność na pękanie badanych stopów świadczy o tym, że plastyczna faza Nb_{ss} wprowadzona do mikrostruktury nie jest wystarczającym sposobem na zwiększenie odporności na pękanie stopów z układu Nb-Ti-Al.

1. Introduction

For many years, the niobium aluminides, especially Nb_3Al, have been considered as very attractive engineering materials. High melting point (2060°C), moderate density (7,26 Mg/m^3) and good high temperature strength of these alloys, predestine them to work at high temperatures [1]. However, despite a number of attractive properties, Nb-Al alloys are not used in a wide range of applications. Before these materials can be of wider use, some of their properties need to be improved. The basic disadvantages of the Nb-Al based alloys which must be overcome are their brittleness and poor fracture toughness at ambient temperature.

One of the ways of improving the ductility and fracture toughness of brittle Nb-Al intermetallics is to reduce their grain size by processing (Mechanical Alloying (MA)) [2], and the other one is to incorporate of a ductile phase into the brittle matrix. This ductile phase may be disordered or ordered solid solution of aluminum and ternary elements in niobium (crystal structure *cI*2 or *cP*2) [3].

In this study titanium was selected as a ternary addition to the Nb-Al alloys. The influence of Ti on microstructure developing during processing and after con-

* AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF METALS ENGINEERING AND INDUSTRIAL COMPUTER SCIENCE, 30-059 KRAKÓW, AL. MICKIEWICZA 30, POLAND

solidation of powders was investigated. Also preliminary mechanical tests were carried out. The results obtained from two alloys are presented in this paper.

2. Material and experimental procedure

Two materials were produced by Mechanical Alloying (MA) of pure Nb, Al and Ti powders in a Szegvari-type laboratory attritor mill. The nominal compositions of the alloys were Nb-15%Ti-15%Al and Nb-22%Ti-15%Al (atomic percent), and were designated as alloy A and B, respectively. The total milling time for the both alloys was 100 h. Powders of both alloys collected after milling were sieved through a mesh and powders smaller than 45 μm were investigated. The samples of sieved powders were annealed at 1000°C for 5 h in vacuum, and the rest of the sieved powders were used for consolidation by hot pressing in the argon atmosphere at a temperature of 1300°C and under a pressure of 25 MPa.

In order to identify the existing phases in the alloys after milling, anealing and consolidation, the X-ray phase analysis was used. The microstructure of powders and consolidated material was investigated by Scanning Electron Microscopy (SEM). Images formed by backscattered electrons providing Z contrast were used for this purpose. The investigation was supplemented by Transmission Electron Microscopy (TEM) of compacted samples. The chemical composition of the constituent phases was determined by energy dispersive spectrometry (EDS). The materials after consolidation were also characterized by hardness and fracture toughness measurements. Fracture toughness was evaluated from microcracks propagating from the corners of Vickers indentations on prepolished surfaces.

3. Results and discussion

Milling of the powder mixture for 100 h results in the refinement of powder particle size. Also the shape of powder particles changed from irregular in the initial powders to spherical. During MA powders are subjected to repeated fracturing and subsequent cold welding. The both processes led to the homogenization of chemical composition of milled powder. An example of a powder particle microstructure after 10 h of milling is shown on Fig. 1. The image was produced by backscattered electrons (BSE) in the SEM utilizing Z contrast. Brighter areas represent regions enriched with the heavier element (Nb) while the darker ones with light elements (Al, Ti). After 100 h of milling such areas are not distinguishable any more. Such a behavior is very typical for mechanical alloying and was also observed by D y m e k et. al. [4, 5] for other mechanically alloyed alloys.

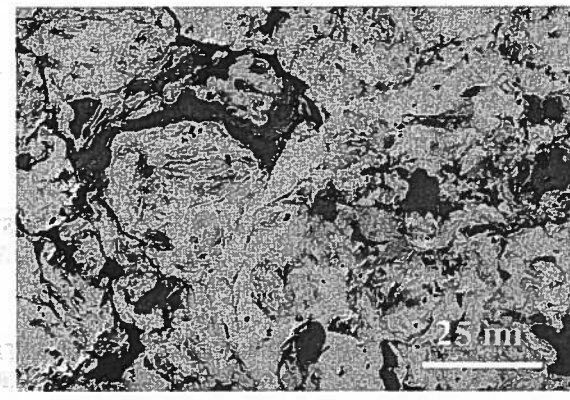

Fig. 1. SEM image of the powder particle (alloy A) after 10 h of milling

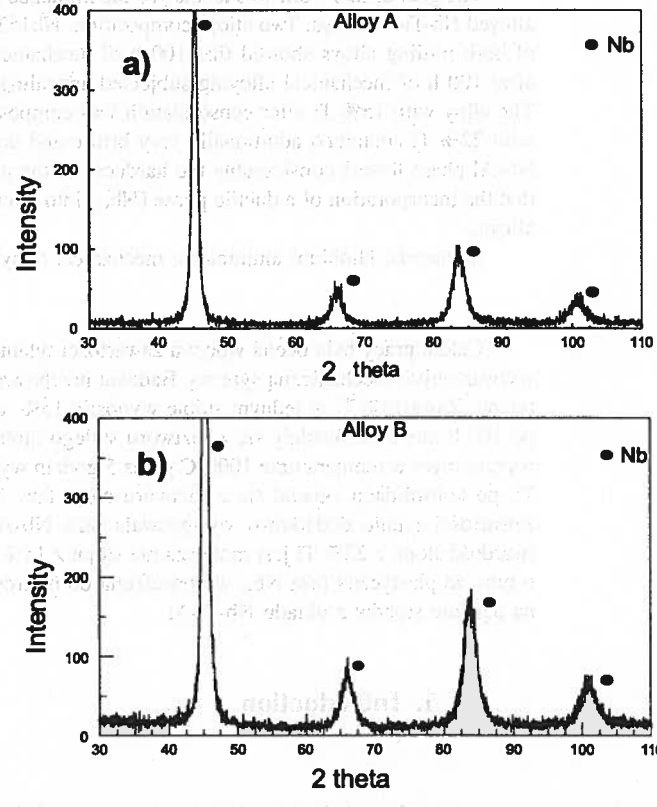

Fig. 2. X-ray diffractograms of the powders after 100 h milling: a) alloy A (CoK_α radiation), b) alloy B (CoK_α radiation)

Fig. 2 presents X-ray diffraction patterns of the powders after 100 h of milling. The X-ray phase analysis showed that after milling, both investigated materials consist only of niobium solid solution Nb_{ss}. Comparing the behavior of alloys *A* and *B* during milling, it could be pointed out that in both alloys Al and Ti dissolve in Nb relatively fast. Peaks from Al and Ti disappeared after 4 h of milling in the alloy *A*. Further milling up to 100 h causes only broadening of Nb peaks. The alloy

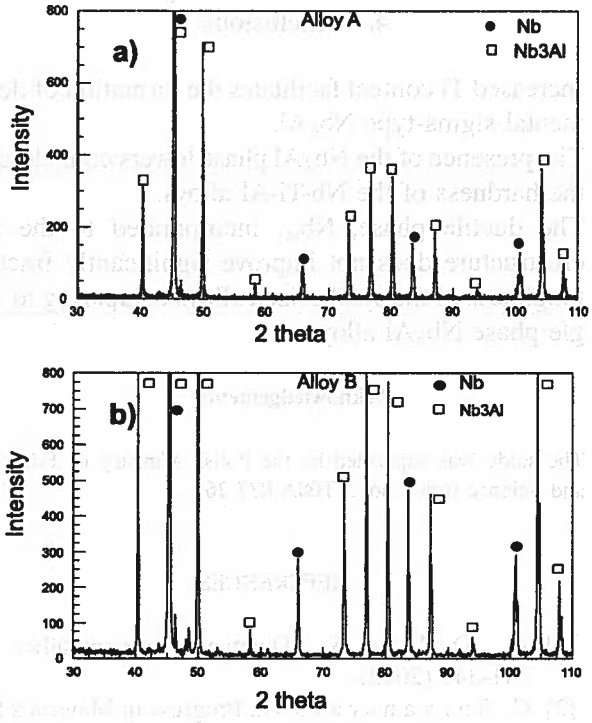

Fig. 3. X-ray diffractograms of the powders after milling for 100 h and annealing at 1000°C for 5 h: a) alloy A (CoKα radiation), b) alloy B (CoKα radiation)

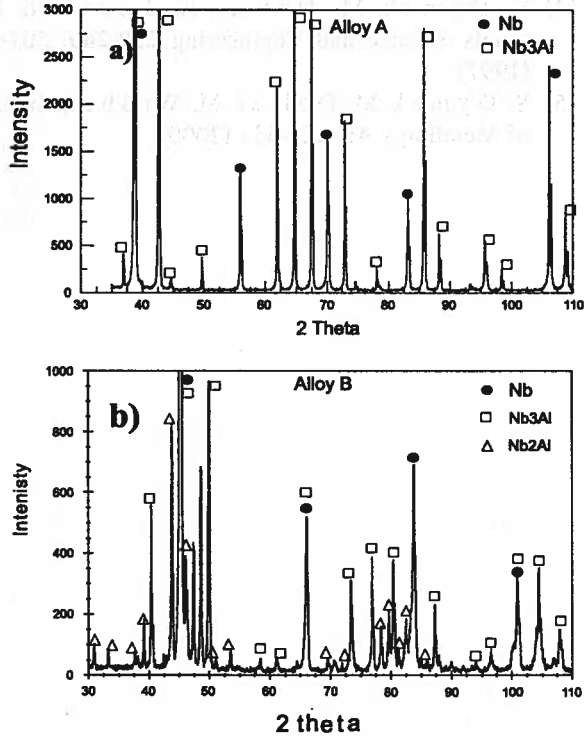

Fig. 4. X-ray diffractograms of the consolidated materials: a) alloy A (CuKα radiation), b) alloy B (CoKα radiation)

with 22% Ti had to be milled for longer time (about 10 h) in order to completely dissolve Al and Ti in the Nb_{ss}. The annealing at 1000°C of the powders milled for 100 h led to the formation of the intermetallic phase Nb3Al

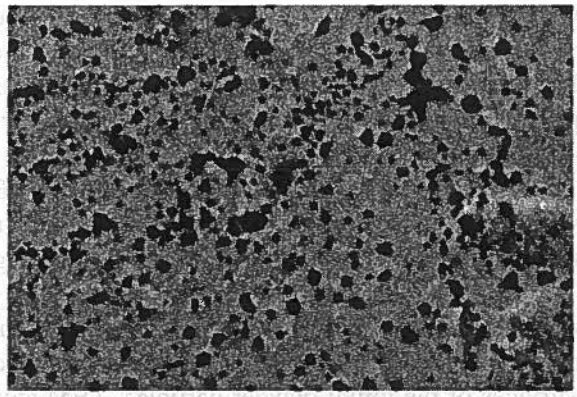

Fig. 5. Backscattered electron image of the Alloy A consolidated at 1300°C

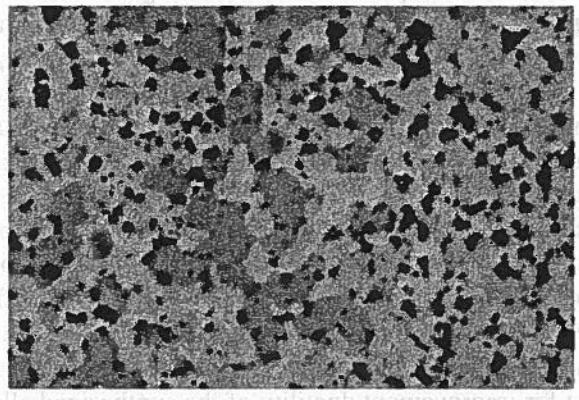

Fig. 6. Backscattered electron image of the Alloy B consolidated at 1300°C

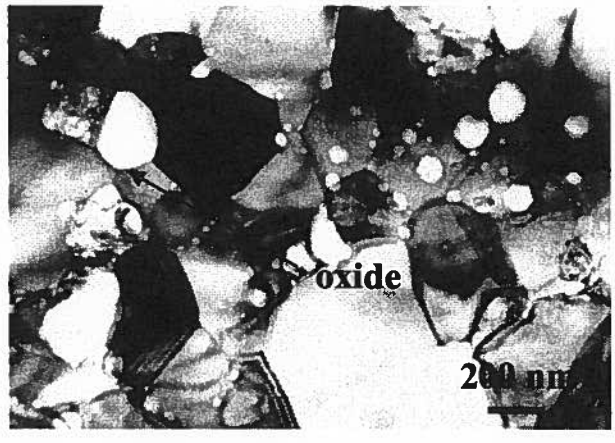

Fig. 7. Micrograph of the alloy A after consolidation, TEM, bright field, acceleration voltage 200 kV

in both alloys (Fig. 3). However, the consolidation of powders at 1300°C brought about formation of different phases in alloys *A* and *B* (Fig. 4). Alloy *A*, like the annealed powder, is composed of Nb_{ss} and Nb_3Al phases while in the alloy *B* strong peaks from the sigma-type Nb_2Al phase were detected by X-ray diffraction. The presence of the Nb_2Al phase is disadventagous. As was

shown by Dollar et al [1] the Nb_2Al phase is detrimental to mechanical properties at room temperature as well lowers creep resistance at elevated temperatures. It is apparent that increased Ti content facilities the formation of this phase.

Scanning electron micrographs formed by BSE showed also an oxide dispersoid which was fairly uniformly distributed in both consolidated alloys (Fig. 5 and 6). Oxides formed as a result of a reaction of Ti and Al with residual oxygen present in the milling chamber. Some oxides, might come from oxide layers which cover the surfaces of the initial powder particles. TEM studies showed that the particles identified on the basis of their chemical composition as oxide dispersoids, were embedded both in other phases or resided on grain boundaries (Fig. 7). TEM studies showed also that the consolidated material exhibited very fine grain size of about 0,5 μm.

The hardness and fracture toughness measurements showed that at 10 kg load, the values of Vickers hardness were 667 and 484 HV, and the values of fracture toughness K_{Ic} were $3,5 \pm 0,2$ MPa·m$^{1/2}$ and $3,4 \pm 0,3$ MPa·m$^{1/2}$ respectively for Alloy *A* and *B*. The lower hardness of alloy *B* is likely associated with the presence of Nb_2Al-type phase. Similar, relatively low, fracture toughness of both alloys suggests that incorporation of a ductile phase (Nb_{ss}) into microstructure is not sufficient for improvement ductility of the synthesized alloys.

Received: 5 January 2006.

4. Conclusions

1. Increased Ti content facilitates the formation of detrimental sigma-type Nb_2Al.
2. The presence of the Nb_2Al phase lowers considerably the hardness of the Nb-Ti-Al alloys.
3. The ductile phase, Nb_{ss}, incorporated to the microstructure does not improve significantly fracture toughness of the synthesized alloys comparing to single phase Nb_3Al alloy.

Acknowledgements

The study was supported by the Polish Ministry of Education and Science (grant no. 3 T08A 037 26).

REFERENCES

[1] A. Dollar, S. Dymek, Intermetallics **11**, 341-349 (2002).
[2] C. Suryanarayana, Progress in Materials Science **46**, 1-184 (2001).
[3] J. Shyue, D-H. Hou, S. C. Johnson, M. Aindow, H. L. Fraser, Materials Research Society **288**, 573-578 (1993).
[4] S. Dymek, M. Dollar, K. Leonard, Materials Science and Engineering **239-240**, 507-514 (1997).
[5] S. Dymek, M. Dollar, M. Wróbel, Archives of Metallurgy **45**, 421-433 (2000).