Intermetallics 42 (2013) 35–40

Contents lists available at SciVerse ScienceDirect

Intermetallics

journal homepage: www.elsevier.com/locate/intermet

Nanocrystalline Al_3Ni_2 alloy with high hardness produced by mechanical alloying and high-pressure hot-pressing consolidation

Marek Krasnowski [a,*], Stanislaw Gierlotka [b], Tadeusz Kulik [a]

[a] Faculty of Materials Science and Engineering, Warsaw University of Technology, Woloska 141, 02-507 Warsaw, Poland
[b] Institute of High Pressure Physics, Polish Academy of Sciences, Sokolowska 29/37, 01-142 Warsaw, Poland

ARTICLE INFO

Article history:
Received 12 March 2013
Received in revised form
18 April 2013
Accepted 27 May 2013
Available online

Keywords:
A. Nanostructured intermetallics
A. Aluminides, miscellaneous
C. Mechanical alloying and milling
C. Powder metallurgy, including consolidation

ABSTRACT

An elemental powder mixture corresponding to Al_3Ni_2 phase stoichiometry was subjected to mechanical alloying. A metastable nanocrystalline AlNi intermetallic phase with the mean crystallite size of 12 nm was formed upon milling. Heating of the synthesised powder in a calorimeter up to 720 °C caused phase transformation into an equilibrium Al_3Ni_2 intermetallic phase with the mean crystallite size of 41 nm. The product of mechanical alloying was consolidated at 1000 °C under the pressure of 5 GPa and 7.7 GPa. During consolidation, a phase transformation analogous with the one observed in the course of heating in the calorimeter took place. Both bulk materials have nanocrystalline structure with mean crystallite size of 67 nm and 58 nm, the smaller one in the sample consolidated under the higher pressure. The hardness of the produced Al_3Ni_2 intermetallic is 8.81 GPa (898 HV1) and 8.72 GPa (887 HV1), while the specific yield strength, estimated using the Tabor relation, is 624 kNm/kg and 617 kNm/kg for the sample hot-pressed under 5 GPa and 7.7 GPa respectively. On the basis of the obtained results, we can assume that the quality of consolidation with preserving a nanocrystalline structure is satisfactory and the hardness as well as the estimated specific yield strength of the produced materials are relatively high.

1. Introduction

Many intermetallic compounds exhibit an attractive combination of physical and mechanical properties [1]. The nickel aluminides intermetallics gain more and more interest and are considered as potential materials for high temperature applications because of their high melting point, high strength and high corrosion and oxidation resistance at elevated temperatures [2]. In the case of intermetallic compounds, where Al constitutes majority of the alloy, low density is an additional advantage.

Melting and casting is a frequently used method for fabrication of nickel aluminides [2]. However, this method has some disadvantages, for example resulting from the large difference between the melting point of Al and Ni or connected with evaporation and oxidation [2,3]. Alternatively, some novel processing techniques, including the Exo-Melt™ process [2], self-propagating high-temperature synthesis (SHS) [4,5] and mechanical alloying (MA) [6,7] have been used to produce nickel aluminides intermetallics. During the MA process, alloys are formed by solid-state reaction. Therefore the MA technique allows to overcome problems such as, e.g., large difference in melting points of the alloying components and evaporation or segregation that could occur during melting and casting. Another advantage of the MA process is that it allows to obtain materials which have a nanocrystalline structure [8,9]. Nanocrystalline materials are potentially attractive for many applications since the reduction of the grain size to the nanometre scale can improve their physical and mechanical properties [10,11]. Some properties, such as high strength and hardness [11–15], that are superior to those of the coarse-grained counterparts, may result from the materials' nanocrystalline structure. There is also some evidence that achieving of nanocrystallinity in intermetallics can lead to improved ductility [16–18].

Nanocrystalline MA products are in the form of powders, however most applications of materials require their bulk forms, so powders should be consolidated after milling. Consolidation of nanocrystalline powders into bulk, full-density material with a nanocrystalline structure preserved is a task both important and difficult to perform. One should note that applying a high temperature during consolidation may lead to grain coarsening, and hence loss of nanocrystalline structure. On the other hand, high temperature is required to obtain good interparticle bonding which provides high-quality consolidation of powders into bulk materials. This is why the serious challenge of compaction processes is to retain nanocrystalline structure of consolidated powders. To fulfil

* Corresponding author. Tel.: +48 22 2348716; fax: +48 22 2348514.
E-mail address: makr@inmat.pw.edu.pl (M. Krasnowski).

0966-9795/$ – see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.intermet.2013.05.017

this, applying a high pressure and limiting the high temperature exposure time during consolidation could be considered. It is shown in the literature that applying the high-pressure hot-pressing technology allows to preserve the nanocrystalline structure during consolidation [13–15,19,20], and we have demonstrated that application of a high pressure influences grain growth at elevated temperature by hindering it [14,15,19]. Considering the grain growth as a diffusional process, this hindering can be explained by the fact that the diffusion coefficient decreases with pressure [21]. Hence, high pressure can reduce grain boundary mobility.

By means of the MA process, a variety of phases, mainly non-equilibrium ones, can be synthesised [9]. Among them, there are supersaturated solid solutions or partially ordered intermetallics [9,22]. With specified chemical composition, supersaturated solid solutions can be considered as disordered intermetallics and upon thermal stimulus their structure orders, thus they transform into ordered intermetallic phases [9,22,23]. Therefore, taking into account the facts mentioned above, mechanical alloying followed by consolidation performed under appropriate conditions can be applied for nanocrystalline nickel aluminides intermetallics manufacturing.

Of the several intermetallic compounds in the nickel-aluminium system, i.e. Al_3Ni, Al_3Ni_2, Al_3Ni_5, NiAl and Ni_3Al, only the last two have been extensively studied [24], while the number of works devoted to Al_3Ni_2 is not very large. Recently, an Al_3Ni_2 intermetallic with the average grain diameter of about 20 μm was produced by SHS synthesis [5]. However, it was concluded that the obtained material contained pores and that the combustion reaction was incomplete, since some residual phases (Ni_3Al, Al_3Ni, Al) were observed in the final product [5]. In this work, a bulk nanocrystalline Al_3Ni_2 intermetallic was produced by high pressure hot-pressing consolidation of mechanically alloyed powders. The structural and phase transformations taking place during the mechanical alloying, during subsequent heating of the milled powder and during consolidation of the final milling product were also studied. The consolidated material was characterised by structural investigations as well as by hardness, density and open porosity measurements. To the best of our knowledge, this is the first production of bulk nanocrystalline Al_3Ni_2 intermetallics.

2. Experimental

An elemental powder mixture of composition 63%Al-37%Ni (all compositions are given in at.% throughout this paper) was mechanically alloyed in a SPEX 8000 D high-energy shaker ball mill. Hardened steel containers and bearing steel balls were used for the milling process. The ball-to-powder weight ratio was 10:1. In order to minimise oxygen contamination, the milling process was performed under a protective argon atmosphere.

The thermal behaviour of the milling product was examined by differential scanning calorimetry (DSC) using a Perkin Elmer DSC7 calorimeter in a temperature range from 50 to 720 °C at a constant heating rate of 40 °C/min.

Powder consolidation was carried out using a press with a high-pressure toroidal chamber. The chamber shape ensured quasi-isostatic pressing conditions. The hot pressing processes were conducted under the pressure of 5 GPa and 7.7 GPa, at the temperature of 1000 °C during 180 s. The loading at the rate of 0.5 GPa/min was done prior to heating. The heating and cooling rate was 1000 °C/min.

The phase changes that occurred in the powder during milling as well as the structure of the material after heating in the calorimeter and after consolidation were investigated by X-ray diffraction (XRD) in a Rigaku MiniFlex II X-ray diffractometer using Cu K_α radiation. To assess the mean crystallite size and the mean lattice strain in powders, the Williamson–Hall method was applied. For calculations from the XRD data, Cu $K_{\alpha1}$ radiation, after $K_{\alpha2}$ stripping using the Rachinger method, was taken into account. The instrumental broadening was determined using an Si standard (provided with the diffractometer), and subtracted from the experimental breadth to obtain the "physical" broadening of each diffraction line which was then used for the Williamson–Hall calculations.

A Zeiss AXIOVERT 40 MAT light microscope and a Hitachi S-3500N scanning electron microscope (SEM) were used for observations of the bulk samples' surface. Samples for SEM and light microscopy were prepared using standard polishing techniques.

The Vickers microhardness and hardness of the compacts were measured using a ZWICK hardness testing device under the load of 200 g and 1 kg respectively, applied for 15 s. The assumed Vickers microhardness and hardness values were the average of at least 25 indentations.

The density of the bulk samples was determined using a Gibertini E154 balance equipped with a device for measuring the density of solids (Archimedes method). The mass measurements performed during density determination allowed to calculate the open porosity of the bulk samples.

3. Results and discussion

The XRD patterns of the powder mixture in the initial state and after selected milling times are shown in Fig. 1. On the basis of these patterns, one can perceive phase transformations occurring in the material during milling. The XRD results reveal that after 2 h of MA a new phase arose in the milled powders. This is demonstrated by a significant drop in the intensity of the Al and Ni diffraction peaks and appearance of new peaks in the diffraction pattern. The new peaks have been attributed to an NiAl phase. After 4 h of MA only peaks corresponding to the NiAl phase are present in the pattern. This phase can be easily formed by MA given its large negative

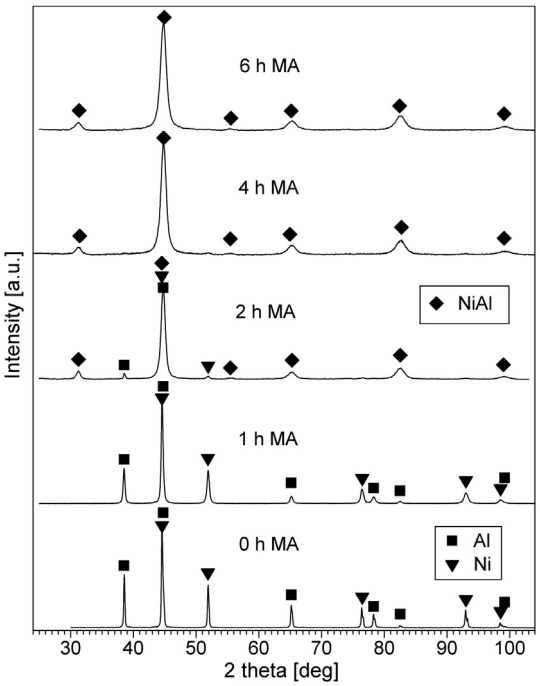

Fig. 1. XRD patterns of $Al_{63}Ni_{37}$ powder mixture mechanically alloyed for the times quoted.