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Journal of Alloys and Compounds 434–435 (2007) 344–347

Journal of ALLOYS AND COMPOUNDS

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Nanocrystalline Ni_3Al alloy produced by mechanical alloying of nickel aluminides and hot-pressing consolidation

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Available online 16 October 2006

Abstract

The reduction of grain size to the nanometer scale can improve the mechanical properties of materials, including intermetallic compounds. In the present study, the mechanical alloying process followed by hot-pressing consolidation has been used to obtain nanocrystalline Ni_3Al intermetallic compound. Nanocrystalline powders of Ni(Al) supersaturated solid solution containing 25 at.% of Al were prepared by ball milling of AlNi or Al_3Ni_2 alloys with addition of Ni powder. Subsequently, the milling products were sintered at 1000 °C under a pressure of 7.7 GPa. The XRD investigations of the consolidated pellets revealed that: (I) ordering of Ni(Al) solid solution and its transformation into a Ni_3Al intermetallic compound occurs during sintering and (II) the material remained nanocrystalline after sintering. The microhardness of Ni_3Al intermetallic produced in this work is of about 980 HV 0.1, the density of compacted materials is nearly 100% of theoretical value and their open porosity is negligible.

Keywords: Intermetallics; Nanostructured materials; Mechanical alloying; Powder metallurgy

1. Introduction

The Ni–Al based intermetallic compounds possess advantageous properties: high tensile strength and yield point, low density, high-temperature creep resistance and good corrosion and oxidation resistance at elevated temperatures [1,2]. Therefore the nickel aluminides are attractive for structural applications at elevated temperatures in hostile environments [1,2]. One of the intermetallics for high-temperatures application is Ni_3Al with the fcc ordered $L1_2$ structure. The strength of the Ni_3Al intermetallic, instead of decreasing with increasing temperature, shows an anomalous increase at intermediate temperatures [3]. Other attractive feature is the fact that its constituent elements are relatively inexpensive. Although there has been a wide research activity in intermetallics in the last years, commercial utilisation of nickel aluminides was hampered because of the low ductility of aluminides and some processing problems (big difference in melting point of Al and Ni and exothermic nature of formation of nickel aluminides) [1,2].

During the mechanical alloying (MA) process alloys are formed by the solid-state reaction [4,5]. Therefore problems such as big difference in melting point of alloying components, segregation or evaporation that could occur during melting and casting can be overcome by using MA process. Usually the MA products possess nanocrystalline or amorphous structure. Nanocrystalline materials are potentially attractive for many applications since the reduction of the grain size to the nanometer scale can improve their physical and mechanical properties [5,6]. In particular, high strength and hardness [7,8] or ductilisation of brittle materials [9,10] superior to conventional materials may be obtained. Furthermore, nanocrystalline structure obtained by milling can improve ductility of intermetallics [11].

The MA process, which can overcome some processing problems, followed by consolidation, could be an alternative for traditional manufacturing processes like melting and casting. Many high temperature melting intermetallics which are difficult to prepare by conventional processing techniques could be easily synthesised with homogeneous structure and composition by the MA process. Consolidation of milled powders into full-density compacts preserving nanometric grain size is not easy to achieve. One of the consolidation methods which allows to retain nanocrystalline structure is hot-pressing [12,13].

The synthesis of alloy with composition Ni–25%Al (at.%) by MA of elemental powder mixtures has been reported in a few works [14–16]. The final product of these processes was Ni(Al) supersaturated solid solution. Recently we obtained nanocrystalline Ni–25%Al alloy with the same structure (Ni(Al)) not by MA of elemental powders, but by milling of AlNi or Al_3Ni_2

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doi:10.1016/j.jallcom.2006.08.209

nickel aluminides with addition of Ni [17]. During heating of milling products in the calorimeter, in both cases, the Ni(Al) solid solution transforms into the ordered Ni_3Al intermetallic compound [17].

The aim of this work was to produce bulk nanocrystalline Ni_3Al intermetallic by hot-pressing consolidation of the mechanically alloyed powders.

2. Experimental

Nanocrystalline powders with stoichiometric Ni_3Al composition were produced by ball milling of powderised AlNi or Al_3Ni_2 intermetallic compounds with addition of Ni powder in appropriate amount. The MA process was performed using a SPEX 8000 D high-energy ball mill, with a steel vials and balls. The details of this experimental step have been described elsewhere [17].

A toroid-type high pressure cell was used for hot-pressing consolidation of the milled powders. The sintering process was performed under a pressure of 7.7 GPa at a temperature of 1000 °C for 180 s.

The X-ray diffraction (XRD) investigations of the milling product and consolidated material were carried out in a Philips 1830 diffractometer using Cu K_α radiation. The lattice parameters, the mean crystallite size and the mean lattice strain, the latter two determined by the Williamson–Hall method, were calculated from the XRD data taking into account Cu $K\alpha_1$ radiation, after $K\alpha_2$ stripping using the Rachinger method. The instrumental broadening was determined using an Si standard (provided with the diffractometer) and subtracted from the experimental breadth to obtain a "pure" broadening of each diffraction line, which was then used for the Williamson–Hall calculations.

A GEMINI LEO1530 scanning electron microscope (SEM) was used for observations of the surface of the hot-pressed sample in order to check for the presence of pores or voids. Samples for SEM observations were prepared using standard polishing techniques.

The average hardness of the compacts was measured using a ZWICK microhardness tester under a load of 100 g imposed for 15 s, and their density using a Gibertini E154 analytical balance equipped with a device for measuring the density of solids (Archimedes method). Basing on mass measurements performed during density determination, open porosity of bulk samples was calculated.

3. Results and discussion

The phase changes occurring in the powders during milling, investigated by XRD, have already been described and analysed in detail [17,18]. It was concluded that for both milled compositions – AlNi + Ni and Al_3Ni_2 + Ni powder mixtures, hereafter referred to as samples A and B – after about 15 h of milling the steady-state stage is reached and that the final product of performed MA processes was nanocrystalline Ni(Al) supersaturated solid solution with a crystallite size of about a dozen nm.

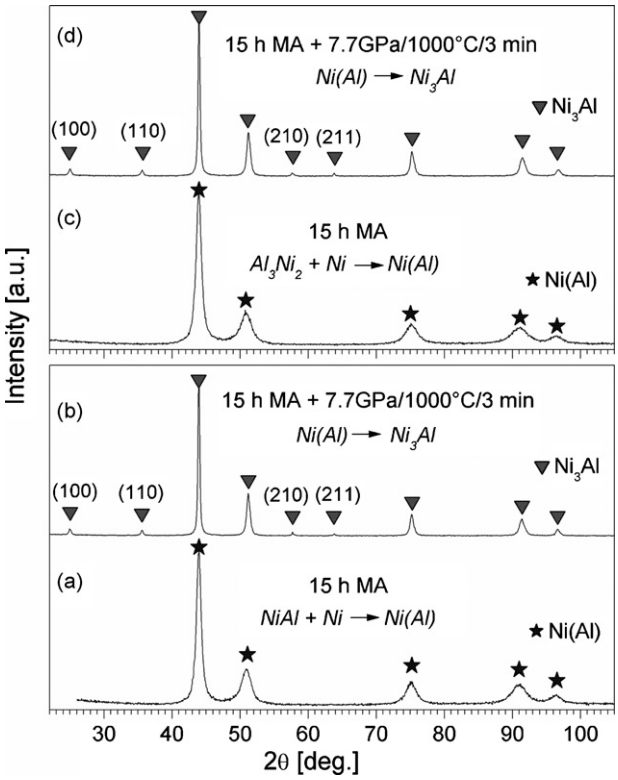

Fig. 1. The XRD patterns of the final MA products before: (a) and (c) and after consolidation: (b) and (d). Lower part of the Fig. 1 relates to AlNi + Ni milled powder mixture and upper part to Al_3Ni_2 + Ni milled powder mixture.

The powders after 15 h of mechanical alloying in A and B milling processes were subjected to hot-pressing consolidation. The XRD patterns of the final MA products before and after consolidation are shown in Fig. 1. Comparing the spectra of bulk samples with those of the powders before hot-pressing, one can see that the (1 0 0), (1 1 1), (2 1 0) and (2 1 1) superlattice reflections of the ordered $L1_2$ structure appear, which evidences the ordering of the Ni(Al) solid solution and its transformation into a Ni_3Al intermetallic. Another feature in these XRD patterns is that the peaks became a little sharper than those in the patterns of the milled powders before consolidation. This reduction in peaks

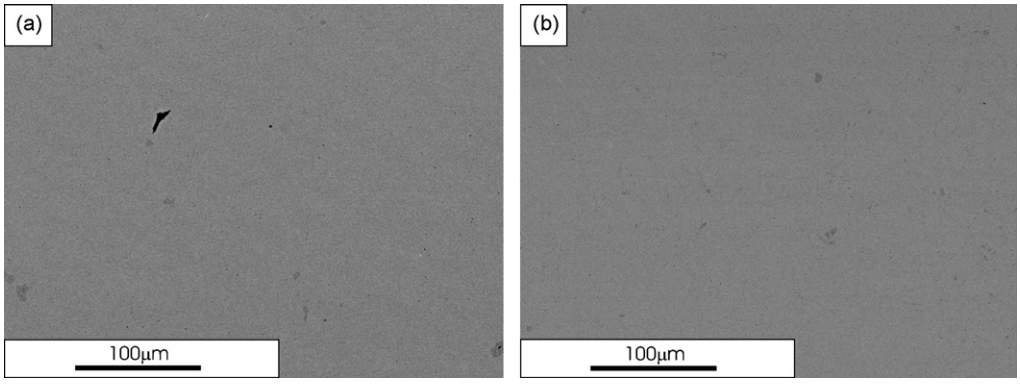

Fig. 2. SEM (backscattered electron (BSE) mode) micrographs of the surface of the polished samples compacted: (a) after A milling process and (b) after B milling process.

Table 1
Characteristics of the bulk samples

	Ni_3Al intermetallic compound obtained from AlNi + Ni mixture	Ni_3Al intermetallic compound obtained from Al_3Ni_2 + Ni mixture	Ni_3Al phase (according to JCPDS PDF card 09-0097)
Lattice parameter (Å)	3.572	3.570	3.572
Mean crystallite size (nm)	23	21	
Mean lattice strain (%)	0.06	0.07	
Microhardness (HV0.1)	981	983	
Density (g/cm^3)	7.39	7.33	7.399
Porosity (%)	∼0	0.2	

width is due to increase of the mean crystallite size and decrease of the mean lattice strain. The mean crystallite size of Ni(Al) for samples A and B before consolidation was about 14 nm. For the compacted samples A and B the mean crystallite sizes of Ni_3Al, determined by the Williamson–Hall formula, are 23 nm and 21 nm, respectively, while the mean lattice strains values are 0.06% and 0.07%, respectively. Hence, nanoscale grain size was retained after applied consolidation process. The lattice parameters of Ni_3Al, calculated from XRD data, for the consolidated samples A and B, are 3.572 Å and 3.570 Å, respectively. These values are in accordance with the lattice parameter of Ni_3Al phase standard, which is equal to 3.572 Å [19].

Fig. 2 shows SEM (backscattered electron (BSE) mode) micrographs of the surface of the polished compacted samples. One can see that the surface is smooth, free of pores or voids, which evidences good quality of consolidation. Since the BSE signal in SEM is sensitive to atomic number, no contrast difference in the Fig. 2 indicates chemical homogeneity of the consolidated samples.

The values of average Vickers microhardness of the Ni_3Al intermetallic for the consolidated samples A and B are nearly the same: 981 HV0.1 with a standard deviation of 19, and 983 HV0.1 with a standard deviation of 18, respectively. For comparison, the microhardness of the Ni_3Al alloy synthesised by powder metallurgy method by a route involving rapid solidification, milling and consolidation by hot isostatic pressing has been found to be 420 HV0.5 [20], obtained via severe plastic deformation (SPD) by torsion (Bridgman method) to be 9200 MPa [21] or nearly 9000 MPa [22], whereas processed by severe rolling at liquid nitrogen temperature to be 660 HV0.1 [23]. On the basis of quoted results, we can assume that the microhardness of the Ni_3Al intermetallic obtained in this work is relatively high.

The density values of the consolidated pellets for the samples A and B are: 7.40 g/cm^3 and 7.33 g/cm^3, respectively, which are 100% and 99.1% of the theoretical density of Ni_3Al phase. The open porosity for the bulk samples A and B is 0% and 0.2%, respectively (say, negligible for both samples).

Characteristics of the bulk samples are summarised in Table 1.

4. Conclusions

Nanocrystalline powder of Ni(Al) containing 25 at.% of Al, produced by mechanical alloying of AlNi + Ni or Al_3Ni_2 + Ni powder mixtures, can be successfully consolidated by hot-pressing at 1000 °C for 180 s under a pressure of 7.7 GPa. Ordering of the Ni(Al) solid solution and its transformation into a Ni_3Al intermetallic compound occurred during the compaction process. Furthermore, limited growth of grains up to a value of about 20 nm took place during the consolidation process. Hence, nanoscale grain size was retained after applied consolidation process and bulk nanocrystalline Ni_3Al intermetallic compound was obtained. The average microhardness of the produced intermetallic is of about 980 HV0.1. The density of compacted materials is nearly 100% of theoretical value and their open porosity is negligible. Observations of the polished samples with the SEM reveal that the surface is smooth, without pores or voids and that produced material is chemically homogeneous.

Acknowledgements

The authors would like to thank Dr. S. Gierlotka (High Pressure Research Center, Polish Academy of Sciences, Warsaw, Poland) for assistance in performing the hot-pressing consolidation.

This work was supported by the State Committee for Scientific Research (grant no. 4 T08A 046 25).

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