Journal of Alloys and Compounds 434–435 (2007) 272–274

Journal of ALLOYS AND COMPOUNDS

www.elsevier.com/locate/jallcom

Fabrication and structure of bulk nanocrystalline Al–Si–Ni–mishmetal alloys

Jerzy Latuch *, Grzegorz Cieslak, Tadeusz Kulik

Faculty of Materials Science and Engineering, Warsaw University of Technology, Wołoska Street 141, 02-507 Warsaw, Poland

Available online 16 October 2006

Abstract

Al-based alloys of structure consisting of nanosized Al crystals, embedded in an amorphous matrix, are interesting for their excellent mechanical properties, exceeding those of the commercial crystalline Al-based alloys. Recently discovered nanocrystalline Al alloys containing silicon (Si), rare earth metal (RE) and late transition metal (Ni), combine high tensile strength and good wear resistance. The aim of this work was to manufacture bulk nanocrystalline alloys from Al–Si–Ni–mishmetal (Mm) system. Bulk nanostructured $Al_{91-x}Si_xNi_7Mm_2$ (x = 10, 11.6, 13 at.%) alloys were produced by ball milling of nanocrystalline ribbons followed by high pressure hot isostating compaction.

Keywords: Nanostructured materials; Rapid-solidification; Sintering

1. Introduction

During the past a few decades a great effort has been devoted to the research of rapidly solidified aluminium alloys. Particularly, multi-component Al–TM (TM, transition metals) and rare earth metal (RE) alloys have been studied intensively. The Al–TM–RE alloys differ qualitatively from the commercially available Al-based alloys (e.g. Al–Si) showing moderate strength levels (up to 300 MPa) and a low thermal stability. Until the discovery of the great potential of rapid solidification the Al-based alloys had been considered to progressively approach their limits. Nanocrystalline aluminium based alloys have gathered much interest, due to their excellent mechanical properties. The melt spinning process is the most popular method in preparing such alloys for scientific research. However, the industrial application of these alloys is limited by the reduced dimensions, obtained by the traditional rapid solidification techniques. That is why, many attempts to produce nanocrystalline aluminium alloys in bulk form have been carried out. Recently discovered nanocrystalline Al alloys containing silicon (Si), rare earth metal (RE) and late transition metal (Ni), combine high tensile strength and good wear resistance [1,2].

The aim of this work was to manufacture bulk nanocrystalline alloys from Al–Si–Ni–Mm system. Bulk nanostructured $Al_{91-x}Si_xNi_7Mm_2$ (x = 10, 11.6, 13 at.%) alloys were produced by ball milling of rapidly solidified ribbons followed by high pressure hot isostating pressing. In respect of the high costs, RE was substituted by an order of magnitude cheaper mishmetal (Mm) of similar properties.

2. Experimental

Master alloys with the composition $Al_{91-x}Si_xNi_7Mm_2$ (x = 10, 11.6, 13 at.%) were prepared from pure elements by arc melting in an argon atmosphere. Rapidly solidified ribbons with a width of 2 mm and thickness of 25 μm were manufactured by melt-spinning technique under argon atmosphere. The melt was ejected onto a copper wheel rotating at peripheral speed of 50 ms^{-1}. Composition of mishmetal (Mm), in at.%, was—Ce: 50.3, La: 43.5, Pr: 5.9 and Nd: 0.3. Resulting ribbons were shiny and with no signs of holes and no rough edges. The ribbons were milled using Fritsch Pulverisette 5 planetary ball mill in argon atmosphere at a ball-to-ribbons weight ratio of 20:1 for 50 min. Different temperatures up to 370 °C and pressure of compaction 7.7 GPa were applied to compact investigated alloys. The dimensions of bulk samples were limited to cylinders of 5 mm in diameter and a maximum of 5 mm in height. Crystallization kinetics was examined by differential scanning calorimeter (DSC) at a scanning rate of 40 K/min. The structures of bulk alloys were characterized by X-ray diffraction (XRD) using Cu Kα radiation and by transmission electron microscopy (TEM). Thin foils were made by electro polishing of 3-mm discs drilled directly from the bulk samples. Scanning electron microscopy (SEM) was used to investigate the quality of compaction of the bulk samples. The Vickers hardness was measured by hardness tester with a load of 1 kG.

* Corresponding author. Tel.: +48 22 6608408; fax: +48 22 6608514.
E-mail addresses: jlat@inmat.pw.edu.pl (J. Latuch), gcieslak@inmat.pw.edu.pl (G. Cieslak), tkulik@inmat.pw.edu.pl (T. Kulik).

0925-8388/$ – see front matter © 2006 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2006.08.148

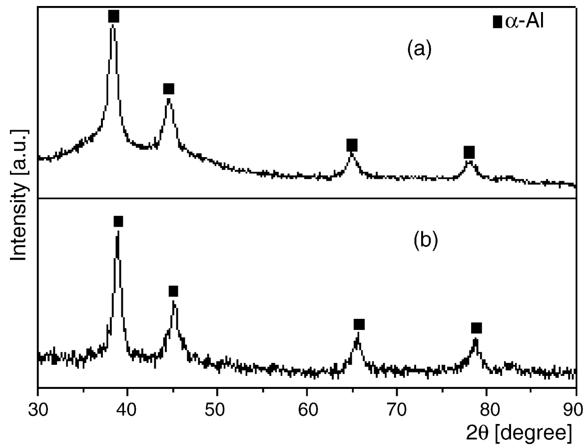

Fig. 1. XRD patterns of as-melt-sun ribbon (a) and bulk (b) $Al_{78}Si_{13}Ni_7Mm_2$ alloy.

3. Results and discusion

Figs. 1–3 Figs. 1a, 2a and 3a show XRD patterns taken from the melt-spun $Al_{78}Si_{13}Ni_7Mm_2$, $Al_{79.4}Si_{11.6}Ni_7Mm_2$ and $Al_{81}Si_{10}Ni_7Mm_2$ ribbons, respectively. As can be seen, not only crystalline peaks corresponding to the α-Al phase are visible but also "halo" related to the amorphous phase. Figs. 1–3 Figs. 1b, 2b and 3b display the XRD traces obtained from the hot compacted alloys. One can see that the microstructure of ribbons and bulk alloys are nearly the same. Calculated lattice parameter of Al-phase (Table 1) is smaller than that of pure Al (0.4047 nm). This phase seems to be a supersaturated solid solution containing rare earth, silicon and nickel elements.

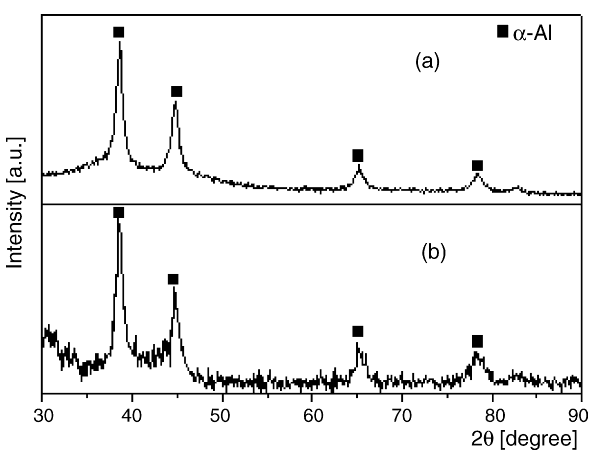

Fig. 2. XRD patterns of as-melt-sun ribbon (a) and bulk (b) $Al_{79.4}Si_{11.6}Ni_7Mm_2$ alloy.

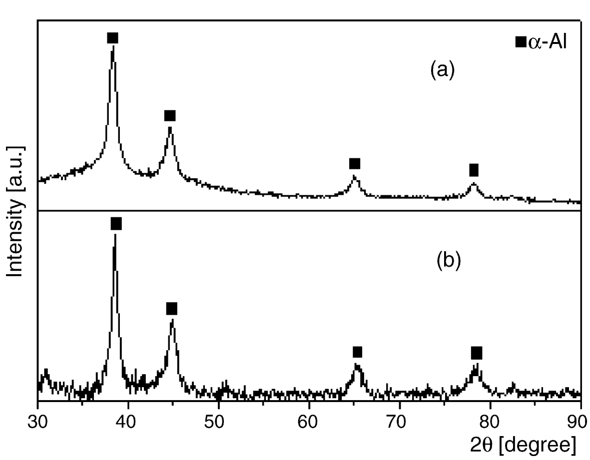

Fig. 3. XRD patterns of as-melt-sun ribbon (a) and bulk (b) $Al_{81}Si_{10}Ni_7Mm_2$ alloy.

Table 1
Hardess value and lattice parameter of invastigated alloys

Alloy	Hardness (HV)	Lattice parameter (nm)
$Al_{78}Si_{13}Ni_7Mm_2$	243	0.4029
$Al_{79.4}Si_{11.6}Ni_7Mm_2$	297	0.404
$Al_{81}Si_{10}Ni_7Mm_2$	317	0.4036

The temperatures of high pressure consolidation were chosen from DSC data obtained for the ribbons. Fig. 4 illustrates DSC curves of hot compacted alloys. The observed, in all investigated alloys, two overlapping peaks correspond to the precipitation mainly of intermetallic compounds. The bulk alloys preserve the nanocrystalline structure up to 270 °C. The microstructure generally consists of randomly distributed fcc-Al nanocrystals,

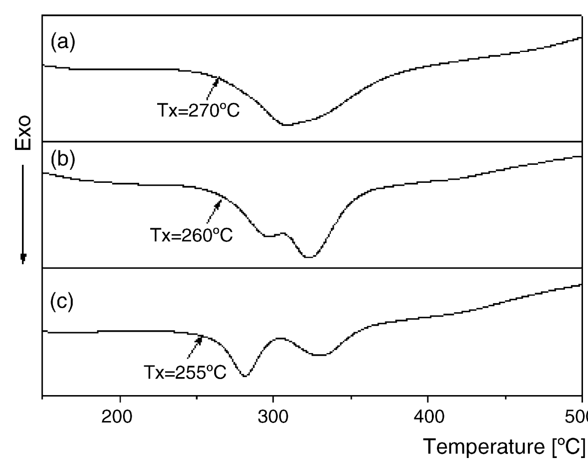

Fig. 4. DSC traces of bulk alloys: $Al_{81}Si_{10}Ni_7Mm_2$ (a), $Al_{79.4}Si_{11.6}Ni_7Mm_2$ (b), $Al_{78}Si_{13}Ni_7Mm_2$ (c).

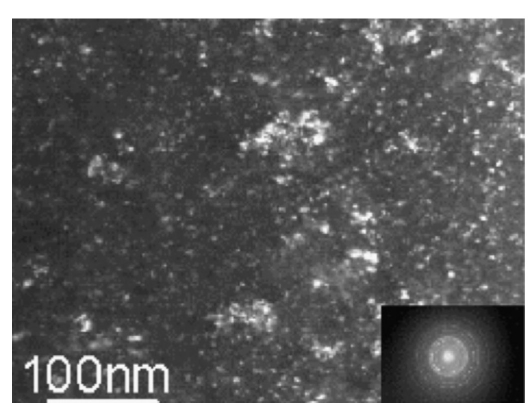

Fig. 5. Dark field image with selected area diffraction pattern of $Al_{78}Si_{13}Ni_7Mm_2$ bulk nanostructured material.

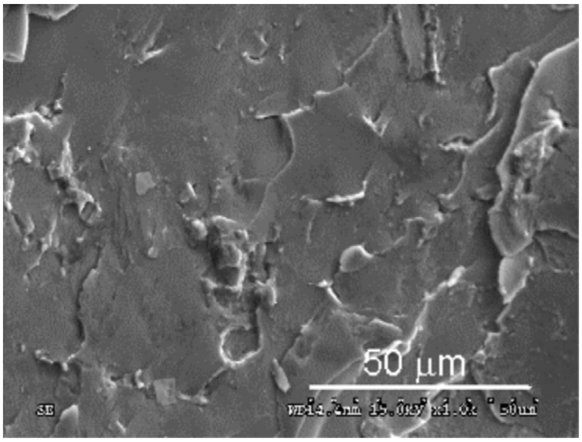

Fig. 6. SEM of fracture surface of $Al_{79.4}Si_{11.6}Ni_7Mm_2$ bulk alloy.

as seen on the TEM image shown on Fig. 5. Nanosized particles spherical in shape and ranging from 10 to 30 nm in size is revealed in the structure. The selected area electron diffraction pattern (Fig. 5) also confirms the nanocrystalline nature of the samples. Figs. 6–8 show the SEM micrographs of the fracture surface of three bulk alloys. Only interparticle fracture is visible which is characteristic for brittle alloys.

The microhardness values obtained in this work (Table 1), with a maximum value of 320 HV, are comparable to the best found for bulk Al–Si–Ni–Ce alloys produced by hot extrusion [2]. However, in that work, these values are achieved for microstructures containing not only amorphous matrix and nanocrystals of α-Al phase but other crystalline phases.

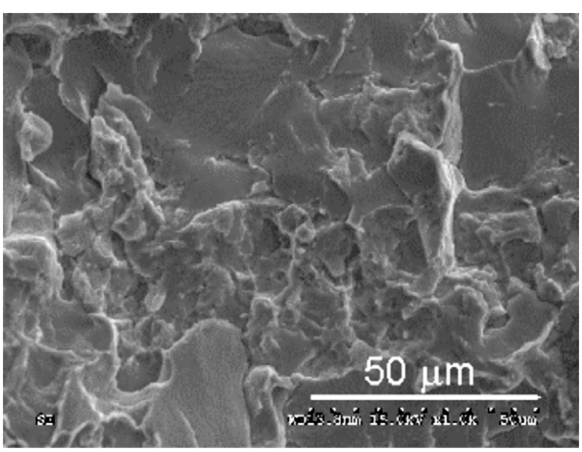

Fig. 7. SEM of fracture surface of $Al_{81}Si_{10}Ni_7Mm_2$ bulk alloy.

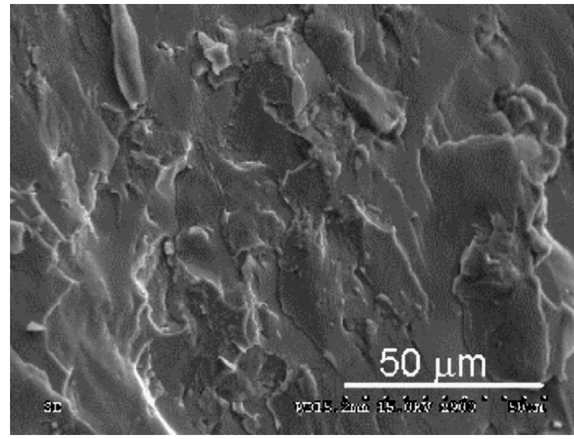

Fig. 8. SEM of fracture surface of $Al_{78}Si_{13}Ni_7Mm_2$ bulk alloy.

4. Conclusions

On the basis of the results reported in this study, the following conclusions can be drawn:

- Bulk $Al_{91-x}Si_xNi_7Mm_2$ (x = 10, 11.6, 13 at.%) alloys were successfully manufactured by ball milling of nanocrystalline ribbons followed by high pressure hot compaction.
- The microstructure of these alloys consist of randomly distributed spherically shaped Al nanoparticles of about 10–30 nm.
- The hardness of bulk nanocrystalline samples depends on aluminium content and achieved values three times higher than that of commercial crystalline Al–Si.

Acknowledgement

The financial support from the Polish Committee of Scientific Research under project PBZ/KBN-096/T08/2003 is gratefully acknowledged.

References

[1] Y. Kawamura, A. Inoue, K. Sasamori, T. Masumoto, Scripta Metall. 29 (1993) 275.
[2] T.H. Lee, Y. Kawamura, A. Inoue, S.S. Cho, T. Masumoto, Scripta Metall. 36 (1997) 475.