Journal of Alloys and Compounds 386 (2005) 182–184

Journal of ALLOYS AND COMPOUNDS

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The isothermal section of the La–Ni–Nb ternary system at 673 K

Huaiying Zhou [a,c,*], Cuiyun He [b], Jingqi Liu [b], Songliu Yuan [a], Jialin Yan [b], Junxia Lü [b]

[a] *Institute of Material Physics and Department of Physics, Huazhong University of Science and Technology, Wuhan, Hubei 430074, PR China*
[b] *Key Laboratory of Nonferrous Metal Materials and New Processing Technology, Guangxi University, Ministry of Education, Nanning, Guangxi 530004, PR China*
[c] *Institute of Materials Science, Guilin University of Electronic Technology, Guilin, Guangxi 541004, PR China*

Received 22 March 2004; received in revised form 4 June 2004; accepted 4 June 2004

Abstract

The isothermal section of the phase diagram of the ternary system La–Ni–Nb at 673 K has been investigated by X-ray diffraction, differential thermal analysis, optical microscopy, and electron microscopy techniques. It consists of 14 single-phase regions, 25 two-phase regions, and 12 three-phases regions. At 673 K, the maximum solid solubilities of Nb in Ni, $LaNi_5$, La_2Ni_7, $LaNi_3$, La_7Ni_{16}, and La_2Ni_3 is about 3, 5, 2, 3, 3, and 2 at.% Nb, respectively. The composition range of the Nb in $NbNi_3$ is about from 23.5 to 27 at.% Nb. No ternary compounds were observed.

Keywords: Phase diagram; X-ray diffraction; La–Ni–Nb system; Isothermal section

1. Introduction

The alloys of the La–Ni system are hydrogen storage materials. Now, the $LaNi_5$ based alloys are being used as metal hydride (MH) electrode for their excellent electrochemical performance. Multi-component La–Ni alloys with a high discharge capacity and long cycle lifetime have been widely investigated.

The phase diagram is an important basis for materials research and material application. The La–Ni phase diagram is very useful for research in hydrogen storage alloys. Some phase diagrams of La–Ni–M ternary system were studied in [10–13], but the phase diagram of La–Ni–Nb has not been published. In this paper, we will report on the results of our investigation of the phase relationship in the 673 K isothermal section of the La–Ni–Nb system. The Ni–Nb binary phase diagram was reported in [6]. Three compounds: $NbNi_8$, $NbNi_3$, and Nb_7Ni_6 were reported in it. Three compounds: NiNb, ζ-$NbNi_4$, and Nb_zNi were reported in [5,8,9], two compounds: Nb_8Ni_{92} and $Nb_{15}Ni_2$ were reported in [7]. Eight kinds of La–Ni compound: La_3Ni, La_7Ni_3, LaNi, La_2Ni_3, $LaNi_2$ (or La_7Ni_{16}), $LaNi_3$, La_2Ni_7, and $LaNi_5$ have been reported in the La–Ni binary system in [1–3]. There is a discrepancy between [1–3] and [10–13] as regards the existence of $LaNi_2$ or La_7Ni_{16}. According to [10–13], La_7Ni_{16} exists instead of $LaNi_2$ and the La_3Ni phase was not observed. The La–Nb binary system phase diagram was reported in [4]. The existence of any compound was not observed in the La–Nb binary system.

2. Experimental details

The present investigation was carried out with 206 samples having masses of about 2 g. The purity of the starting elements were 99.9% La, 99.99% Ni, and 99.99% Nb, respectively. The samples were prepared by arc melting on a water-cooled copper crucible with a non-consumable tungsten electrode under pure argon atmosphere. The samples were re-melted three times for homogeneity. For these samples, the weight loss is less than 1% after melting. Therefore, no chemical analyses were carried out.

After melting, the samples were sealed in an evacuated quartz tube for homogenization annealing. The heat treatment temperature was chosen according to the phase

* Corresponding author.
E-mail address: zhuang@gxu.edu.cn (H. Zhou).

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doi:10.1016/j.jallcom.2004.06.017

diagram of the binary La–Ni and Nb–Ni system. The samples in the La-rich part and the Ni-rich part were annealed at 773 K for 720 h. The others were annealed at 1073 K for 480 h. Then, all these samples were cooled down to 673 K at a rate of 0.15 K/min and kept at 673 K for 240 h. In the end, the samples were quenched into liquid nitrogen.

All these homogenized alloy powders or buttons were investigated by X-ray diffraction, which was carried out on a Rigaku D/Max 2500v powder diffractometer equipped with Cu Kα radiation and graphite monochromator which was operated at 40 kV and 200 mA. The analysis software is Jade5.0 [8] and the 2θ scan range was from 20 to 60°. Some typical alloys were analyzed in an S-570 scanning electron microscope (SEM), by electron probe microanalysis (EPMA) or differential thermal analysis (DTA). From all these data, the phase relations in the La–Ni–Nb system were determined.

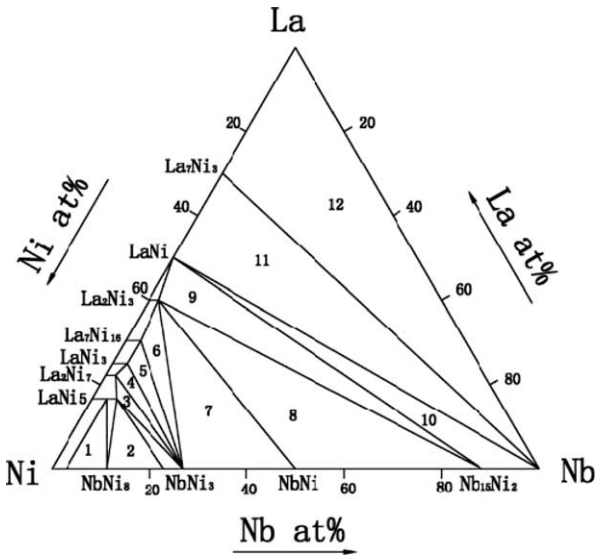

Fig. 1. The isothermal section of the La–Ni–Nb ternary system at 673 K.

3. Results and discussion

3.1. Binary system

3.1.1. La–Ni system

In our investigation, seven compounds: La_7Ni_3, LaNi, La_2Ni_3, La_7Ni_{16}, $LaNi_3$, La_2Ni_7, and $LaNi_5$ were confirmed. We did not observe La_3Ni. The XRD pattern of the sample of "La_3Ni" in our experiment consisted of the patterns of La_7Ni_3 and La.

3.1.2. Ni–Nb system

Four kinds of Ni–Nb compounds: $NbNi_8$, $NbNi_3$, NbNi, and $Nb_{15}Ni_2$ were confirmed in our experiment. The Nb_zNi compound ($z = 5 \pm x$, 96F, $Fd3m$, $a = 1.1642$ nm, $NiTi_2$ structure type) was first reported in [5] and it was later expressed as $Nb_{15}Ni_2$ (cF96, $Fd\bar{3}m$, $a = 1.1642$ nm, $NiTi_2$ structure type) in [7]. The $Nb_{15}Ni_2$ compound was not reported in the Nb–Ni binary phase diagram [6], but we observed the pattern of $Nb_{15}Ni_2$ (Nb_zNi) for 88.2% Nb, 11.8% Ni, and several other corresponding regions in our experiment. So we consider that the $Nb_{15}Ni_2$ compound exist under our experimental conditions. Otherwise, we did not observe the Nb_8Ni_{92} and the ζ-$NbNi_4$ compounds in our investigation. The patterns of "ζ-$NbNi_4$" and several alloys near "ζ-$NbNi_4$" consisted of $NbNi_8$ and $NbNi_3$.

3.1.3. La–Nb system

By comparing and analyzing the X-ray diffraction patterns of the samples in the La–Nb system, no binary compound was observed.

3.2. Solid solubility

At 673 K, the maximum solid solubility of Nb in Ni, $LaNi_5$, La_2Ni_7, $LaNi_3$, La_7Ni_{16}, and La_2Ni_3 is about 3, 5, 2, 3, 3, and 2 at.% Nb, respectively. The concentration of Nb in $NbNi_3$ ranges from 23.5 to 27 at.% Nb. The solid solubility in the other single-phase regions is too small to be observed.

3.3. Isothermal sections

By comparing and analyzing the X-ray diffraction patterns of 206 samples, the isothermal section of the La–Ni–Nb ternary system at 673 K was determined, as shown in Fig. 1. The 673 K isothermal section consists of 14 single-phase regions, 25 two-phase regions, and 12 three-phase regions. The 14 single-phase region are La, La_7Ni_3, LaNi, La_2Ni_3, La_7Ni_{16}, $LaNi_3$, La_2Ni_7, $LaNi_5$, Ni, $NbNi_8$, $NbNi_3$, NbNi, $Nb_{15}Ni_2$, and Nb. Details of the three-phase regions are given in Table 1. No ternary compound was discovered in the La–Ni–Nb system.

Table 1
Three-phase region of the La–Ni–Nb ternary system

Region number	Phase 1	Phase 2	Phase 3
1	Ni	$NbNi_8$	$LaNi_5$
2	$NbNi_3$	$NbNi_8$	$LaNi_5$
3	$NbNi_3$	La_2Ni_7	$LaNi_5$
4	$NbNi_3$	La_2Ni_7	$LaNi_3$
5	$NbNi_3$	$LaNi_3$	La_7Ni_{16}
6	$NbNi_3$	La_2Ni_3	La_7Ni_{16}
7	$NbNi_3$	La_2Ni_3	NbNi
8	NbNi	La_2Ni_3	$Nb_{15}Ni_2$
9	LaNi	La_2Ni_3	$Nb_{15}Ni_2$
10	LaNi	$Nb_{15}Ni_2$	Nb
11	LaNi	La_7Ni_3	Nb
12	La	La_7Ni_3	Nb

Acknowledgements

This work was jointly supported by the National Natural Science Foundation of China (authorized number: 50001002) and by the Natural Science Foundation of Guangxi (authorized number: 0135005).

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