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Investigation on the isothermal section of the Gd-Ni-Y ternary system at 773 K

Wei He*, Wei Zheng, Jianlong Yang, Lingmin Zeng

Key Laboratory of Nonferrous Metal Materials and New Processing Technology, College of Materials Science and Engineering, Ministry of Education (Guangxi University), Nanning, 530004, PR China

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ABSTRACT

The isothermal section of the phase diagram of the Gd–Ni–Y ternary system at 773 K was investigated by means of X-ray powder diffraction, metallographic analysis and scanning electron microscopy with energy dispersive analysis. The isothermal section consists of 12 single-phase regions, 15 two-phase regions and 4 three-phase regions. Six pairs of corresponding compounds in the Gd–Ni and Y–Ni systems, i.e., Gd₂Ni₁₇ and Y₂Ni₁₇, GdNi₅ and YNi₅, Gd₂Ni₇ and Y₂Ni₇, GdNi₃ and YNi₃, GdNi₂ and YNi₂, Gd₃Ni and Y₃Ni and metals Gd and Y form a continuous series of solid solutions, respectively. At 773 K, the maximum solubilities of Gd in YNi and Y in GdNi were about 20 at.% Gd and 8 at.% Y, respectively. No solubility of Gd in YNi₄ and Y in GdNi₄ was observed.

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1. Introduction

Recently, magnetic refrigeration (MR), based on the magnetocaloric effect (MCE), has been an attractive technology for the low-temperature generation for its high thermodynamic efficiency and environmental safety in comparison with the vapor compression [1]. The search for working substances in the cryogenic temperature range of interest is strongly desired for further improvement of magnetic refrigeration. In recent years, the Gd-Ni intermetallic compounds, such as Gd₃Ni, GdNi₂, have been extensively studied and attracted considerable attention due to their intrinsic magnetic structures and transport properties as well as potential applications to magnetic refrigeration technology. For instance, Gd₃Ni is antiferromagnet and undergoes field-induced metamagnetic transition in the antiferromagnetic phase below its Neel temperature ($T_N = 99 \,\mathrm{K}$). The maximum values of isothermal magnetic entropy change are 18.5 J kg⁻¹ K⁻¹ and 5 J kg⁻¹ K⁻¹ for Gd_3Ni for a field change (ΔH) of 100 kOe and 50 kOe near T_N = 99 K, respectively [2]. In addition, the magnetic structure of Laves phase GdNi₂ with the cubic MgCu₂-type structure reported in terms of a two-sublattice model by Yano et al. [3] is ferromagnetic ($T_C = 85 \text{ K}$) and per Ni atom in GdNi2 retains a magnetic moment of about $0.24 \,\mu_B$, coupled antiparallel to the Gd moment.

Phase diagram is an important basis for materials research and materials application. The binary systems of Gd-Ni, Y-Ni and Gd-Y have been widely investigated and the phase diagrams of these systems were given in Ref. [4]. The Gd-Ni binary phase diagram was first proposed by Copeland et al. [5] and later revised by Novy et al. [6] and Pan et al. [7]. In 1996, Xia and Jin optimized this system [8] by using the CALPHAD technique. It was reported that nine intermetallic compounds exist in the Gd-Ni binary system, i.e., Gd₂Ni₁₇, GdNi₅, GdNi₄, Gd₂Ni₇, GdNi₃, GdNi₂, GdNi, Gd₃Ni₂ and Gd₃Ni in Ref. [7], while Copeland et al. [5] claimed only seven compounds observed in the system. They did not observe the compounds of Gd₃Ni₂ and GdNi₄ but reported the compound Gd₂Ni₁₅ instead of Gd₂Ni₁₇. In Ref. [4], nine binary compounds namely Y₂Ni₁₇, YNi₅, YNi₄, Y₂Ni₇, YNi₃, YNi₂, YNi, Y₃Ni₂ and Y₃Ni of the Y-Ni binary system were reported. According to the phase diagram of Gd-Y binary system, Gd and Y atoms can replace each other and form continuous solid solutions without any intermetallic compounds existing. In Ref. [9], it was reported that there existed two monoclinic ternary compounds Gd₃Ni₄Y and Gd₇Ni₁₀Y₃ with space group of $P2_1/m$ in the Gd-Ni-Y ternary system. The quasi-binary phases Gd_3Ni_4Y with base-structure stacking h_2c_3 and $Gd_7Ni_{10}Y_3$ with base-structure stacking h_2c were formed via mixed stacking variants of the CrB and FeB types in the quasi-binary system GdNi-YNi [10]. According to Ref. [10], the samples of the two quasi-binary phases Gd₃Ni₄Y and Gd₇Ni₁₀Y₃ were held at 1073 K over a period of five weeks followed by slow cooling in the furnace in order to obtain the two phases. In order to provide a basis for searching for new rare earth magnetic materials and improving the performance of materials, investigation on the phase relationship of the

^{*} Corresponding author at: College of Materials Science, Guangxi University, Nanning, Guangxi, 530004, PR China. Tel.: +86 771 327 5918; fax: +86 771 323 9406. E-mail address: wei_he@gxu.edu.cn (W. He).

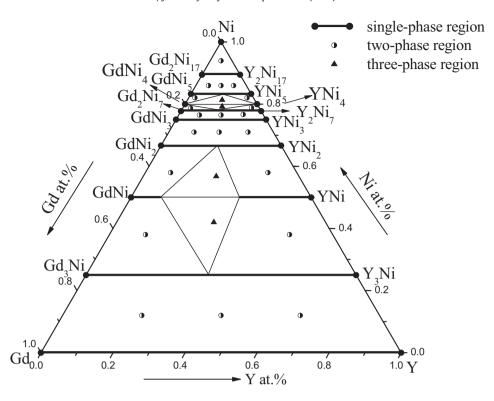


Fig. 1. The isothermal section of the Gd-Ni-Y ternary system at 773 K.

Gd-Ni-RE (rare earth) system is needed. The purpose of this work is to study systematically the phase relation in the Gd-Ni-Y ternary system at 773 K.

2. Experimental details

Samples with 1.5 g or 2 g each were prepared by arc-melting of initial materials of at least of 99.9 wt.% purity under high-purity argon atmosphere. The samples were turned over and remelted three times to ensure good homogeneity. Weight losses during arc melting were less than 1 wt.%. The homogenizing annealing was carried out in an evacuated quartz tube. The Ni-rich alloys were homogenized at 1173 K

for 30 days and the samples which contained less than 50 at.% Ni were annealed at 873 K for 50 days. The other alloys were held at 1073 K for 40 days. After annealing, the samples were cooled down slowly to 773 K and kept for 10 days. Finally, the alloys were quenched into liquid nitrogen. The samples were break into two parts. One parts of the samples for X-ray diffraction analysis were ground into powder and annealed in a small evacuated glass tube at 773 K for 2 days and then quenched into liquid nitrogen. The other parts of the samples were reserved for other analysis when needed. For the metallographic analysis of the samples, standard techniques were used. The metallographic samples were etched by clean water.

The X-ray diffraction data were collected on a Rigaku D/max 2500 V diffractometer with Cu K α radiation and graphite monochromator operated at 40 kV and 200 mA. Phase analysis of the samples was performed by using the computer soft-

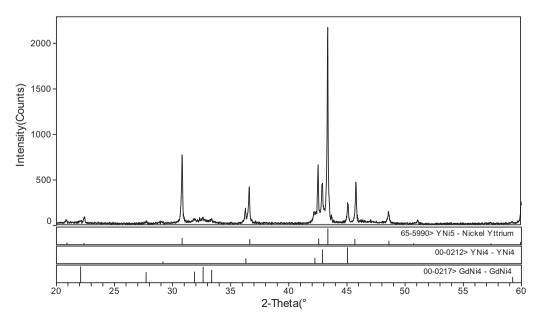
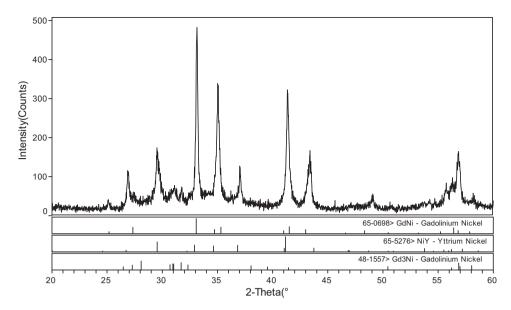


Fig. 2. The X-ray diffraction patterns of sample no. A18 ($Gd_{11}Ni_{81}Y_8$) in the three-phase region of $Gd_{1-x}Y_xNi_5$ (x = 0.5) + $GdNi_4$ + YNi_4 .



 $\textbf{Fig. 3.} \ \ The \ X-ray \ diffraction \ patterns \ of the \ sample \ no. \ C9 \ (Gd_{36.5}Ni_{45.5}Y_{18}) \ located \ in the \ three-phase \ region \ of \ Gd_{3-x}Y_xNi \ (x=1.36) + Gd_{1-x}Y_xNi \ (x=0.16) + Gd_yY_{1-y}Ni \ (x=0.4).$

ware Jade 5.0 [11] applied to the JCPDS-ICDD Powder Diffraction File database (ICDD, 2002 release). Scanning electron microscopy (SEM, Hitachi S-3400 N) with energy dispersive analysis (EDS, EDAX) and optical microscopy were used for microstructural analysis.

3. Results and discussion

3.1. Isothermal section of the Gd-Ni-Y system at 773 K

By comparing and analyzing the X-ray diffraction patterns of 142 binary and ternary alloy samples combined with the results of scanning electron microscopy with energy dispersive analysis and optical microscopy and by identifying the phases in each sample, the 773 K isothermal section of the phase diagram of the Gd–Ni–Y ternary system was determined, shown in Fig. 1. The isothermal section consists of 12 single-phase regions, 15 two-phase regions, and 4 three-phase regions. They are as follows:

12 single-phase regions: Ni, $Gd_{2-x}Y_xNi_{17}$, $Gd_{1-x}Y_xNi_{5}$, $GdNi_{4}$, YNi_{4} , $Gd_{2-x}Y_xNi_{7}$, $Gd_{1-x}Y_xNi_{3}$, $Gd_{1-x}Y_xNi_{2}$, $Gd_{1-x}Y_xNi$ (x = 0 - 0.16), $Gd_{y}Y_{1-y}Ni$ (y = 0 - 0.4), $Gd_{3-x}Y_xNi$, $Gd_{1-x}Y_x$.

 $\begin{array}{lll} 4 & \text{three-phase} & \text{regions:} & \text{Gd}_{1-x}Y_x\text{Ni}_5 & (x=0.5) + \text{GdNi}_4 + \text{YNi}_4, \\ \text{Gd}_{2-x}Y_x\text{Ni}_7 & (x=1.1) + \text{GdNi}_4 + \text{YNi}_4, & \text{Gd}_{1-x}Y_x\text{Ni}_2 \\ (x=0.44) + \text{Gd}_{1-x}Y_x\text{Ni} & (x=0.16) + \text{Gd}_yY_{1-y}\text{Ni} & (y=0.4), & \text{Gd}_{3-x}Y_x\text{Ni} \\ (x=1.36) + \text{Gd}_{1-x}Y_x\text{Ni} & (x=0.16) + \text{Gd}_yY_{1-y}\text{Ni} & (x=0.4). \end{array}$

Fig. 2 presents the X-ray diffraction patterns of sample no. A18 ($\mathrm{Gd}_{11}\mathrm{Ni}_{81}\mathrm{Y}_8$) consisting of the three phases of $\mathrm{Gd}_{1-x}\mathrm{Y}_x\mathrm{Ni}_5$ (x=0.5), GdNi_4 and YNi_4 showing the existence of the three-phase region of $\mathrm{Gd}_{1-x}\mathrm{Y}_x\mathrm{Ni}_5$ (x=0.5)+ GdNi_4 + YNi_4 . Fig. 3 shows the X-ray diffraction patterns of the sample no. C9 ($\mathrm{Gd}_{36.5}\mathrm{Ni}_{45.5}\mathrm{Y}_{18}$) located in the three-phase region of $\mathrm{Gd}_{3-x}\mathrm{Y}_x\mathrm{Ni}$ (x=1.36)+ $\mathrm{Gd}_{1-x}\mathrm{Y}_x\mathrm{Ni}$ (x=0.16)+ $\mathrm{Gd}_y\mathrm{Y}_{1-y}\mathrm{Ni}$ (x=0.4). The SEM micrograph of the sample no. B3 ($\mathrm{Gd}_{28}\mathrm{Ni}_{54}\mathrm{Y}_{18}$) in the three-phase region of $\mathrm{Gd}_{1-x}\mathrm{Y}_x\mathrm{Ni}_2$ (x=0.44)+ $\mathrm{Gd}_{1-x}\mathrm{Y}_x\mathrm{Ni}$ (x=0.16)+ $\mathrm{Gd}_y\mathrm{Y}_{1-y}\mathrm{Ni}$ (y=0.4), as shown in Fig. 4, proving the existence of the phase region. The white gray

background is the phase of $Gd_{1-x}Y_xNi$, the gray area is $Gd_{1-x}Y_xNi_2$ and the small black pieces are $Gd_yY_{1-y}Ni$.

3.2. Phase analysis and solid solubility

Sixteen binary compounds in the Gd–Ni–Y ternary systems have been confirmed to exist at 773 K by analyzing the X-ray diffraction patterns of the binary and ternary samples. The X-ray diffraction patterns of these compounds basically corresponded with the respective PDF data or the diffraction patterns calculated from the crystallographic data available in literature by using the PowderCell program. The binary compounds Gd₂Ni₁₇, GdNi₅, GdNi₄, Gd₂Ni₇, GdNi₃, GdNi₂, GdNi, Gd₃Ni, Y₂Ni₁₇, YNi₅, YNi₄, Y₂Ni₇, YNi₃, YNi₂, YNi, Y₃Ni were found in our binary and ternary samples. No new binary or ternary compound was found in all alloy samples at 773 K.

In order to verify the existence of Gd_3Ni_2 at 773 K, a total of six alloy samples near the composition of Gd_3Ni_2 were prepared. It was found that the X-ray diffraction patterns of each alloy sample

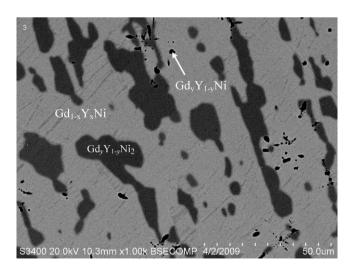


Fig. 4. The SEM micrograph of sample no. B3 ($Gd_{28}Ni_{54}Y_{18}$) in the three-phase region of $Gd_{1-x}Y_xNi_2$ (x=0.44) + $Gd_{1-x}Y_xNi$ (x=0.16) + $Gd_yY_{1-y}Ni$ (y=0.4). White gray background area is the phase of $Gd_{1-x}Y_xNi$, the gray area is $Gd_{1-x}Y_xNi_2$ and the small black pieces are $Gd_yY_{1-y}Ni$.

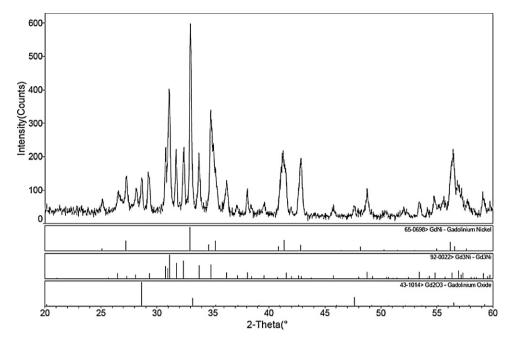


Fig. 5. The X-ray diffraction pattern of the sample no. C6 (Gd₆₀Ni₄₀) containing the two phases of Gd₃Ni and GdNi. (The sample contains a little amount of Gd₂O₃.).

consisted of the diffraction lines of GdNi and Gd_3Ni . No diffraction reflections belong to the phase Gd_3Ni_2 were observed in these samples. No evidence was found to confirm the existence of compound Gd_3Ni_2 at 773 K. This is in good agreement with Ref. [5]. The X-ray diffraction patterns of a selected sample of no. C6 with the composition of Gd_6Ni_{40} consisted of Gd_3Ni and GdNi indicating the absence of the Gd_3Ni_2 phase, as shown in Fig. 5. (The sample contains a little amount of Gd_2O_3).

Similar analysis was carried out to verify the existence of Y_3Ni_2 . The X-ray diffraction patterns of six prepared samples near the composition of Y_3Ni_2 proved that all the alloy samples contained YNi and Y_3Ni . No evidence was found to support the existence of Y_3Ni_2 in our present study. The X-ray diffraction patterns of a typical alloy sample no. C5 ($Ni_{40}Y_{60}$) pointed to that the sample consisted of the two phases of YNi and Y_3Ni and indicated the absence of Y_3Ni_2 at 773 K, seen in Fig. 6. (The sample contains a little amount of Y_2O_3 .)

In order to find out the existence of the compound $GdNi_4$, eight alloy samples with the composition near $GdNi_4$ were prepared in our work. As reported in Ref. [7], it was difficult to obtain the single phase of $GdNi_4$, even after annealing at high temperature (at 1173 K) for a long time (30 d). The X-ray diffraction analysis showed that the sample no. A20 ($Gd_{20}Ni_{80}$) containing the phases of $GdNi_5$, Gd_2Ni_7 and $GdNi_4$ and proving the existence of the compound $GdNi_4$, as shown in Fig. 7. Analysis on the ternary samples with the compositions near $GdNi_4$ gave the similar results.

A total of six samples with the composition near YNi₄ were prepared to identify the existence of YNi₄. The X-ray diffraction analysis shows that all these six samples consist of phases of $Gd_{1-x}Y_xNi_5$ and YNi₄. Similar to the phase of GdNi₄, it was difficult to obtain the single phase of YNi₄. This is because that the peritectic reaction for YNi₄ occurred at about 1613 K and was close to the congruent melting temperature of YNi₅ [4]. The X-ray diffraction patterns of the sample no. A18 ($Gd_{11}Ni_{81}Y_8$) contained the three-phases of $Gd_{1-x}Y_xNi_5$ (x=0.5), $GdNi_4$ and YNi_4 showing the existence of the phases of $GdNi_4$ and YNi_4 , seen in Fig. 2.

In order to determined the three-phase region boundaries of $Gd_{1-x}Y_xNi_2$ (x=0.44) + $Gd_{1-x}Y_xNi$ (x=0.16) + $Gd_yY_{1-y}Ni$ (y=0.4), a series samples with compositions of $Gd_{46-x}Y_xNi_{54}$ were prepared

and analysis by X-ray diffraction and/or electron microscopy with energy dispersive analysis. The X-ray diffraction analysis of these samples pointed out that the phase $Gd_yY_{1-y}Ni$ (y = 0.4) appeared in the sample no. B43 with compositions of $Gd_{46-x}Y_xNi_{54}$ (x = 11.5) indicating the sample fell in the three-phase region of $Gd_{1-x}Y_xNi_2$ $(x=0.44) + Gd_{1-x}Y_xNi \ (x=0.16) + Gd_yY_{1-y}Ni \ (y=0.4)$. The analysis on the variations of the lattice parameters of the phase $Gd_{1-x}Y_xNi$ in the samples $Gd_{46-x}Y_xNi_{54}$ also showed that the lattice parameters of the phase $Gd_{1-x}Y_xNi$ in the samples with compositions of $Gd_{46-x}Y_xNi_{54}$ (11.3 $\leq x \leq$ 18.0) kept unchanged. This means that the samples of $Gd_{46-x}Y_xNi_{54}$ (11.3 $\leq x \leq$ 18.0) were located in the three-phase region, since the lattice parameters and composition of each phase in three-phase region keep unchanged. Thus the left boundary of the three-phase region $Gd_{1-x}Y_xNi_2$ $(x = 0.44) + Gd_{1-x}Y_xNi(x = 0.16) + Gd_yY_{1-y}Ni(y = 0.4)$ is determined. The electron microscopy with energy dispersive analysis also gave the same results. Similarly, the right boundary of this three-phase region was determined.

By similar procedures, the phase boundaries of $Gd_{2-x}Y_xNi_7(x=1.1)+GdNi_4+YNi_4$, $Gd_{1-x}Y_xNi_5$ $(x=0.5)+GdNi_4+YNi_4$ and $Gd_{3-x}Y_xNi$ $(x=1.36)+Gd_{1-x}Y_xNi$ $(x=0.16)+Gd_yY_{1-y}Ni$ (x=0.4) were obtained.

In Ref. [9], the compound Gd_2Ni_{15} was reported instead of the existence of Gd_2Ni_{17} . By comparing the experimental X-ray diffraction data and lattice parameters of the samples with composition near Gd_2Ni_{17} with those of Gd_2Ni_{15} available in JCPDS-PDF file and calculated from the crystallographic data [12] by LAZY program, it was found that Gd_2Ni_{15} is isostructure with the compound Gd_2Ni_{17} with absence of some Ni atoms. This is in agreement with Ref. [5].

Two samples with the composition of Gd_3Ni_4Y and $Gd_7Ni_{10}Y_3$ were prepared in order to verify their existence. The X-ray diffraction analysis showed that the sample with the composition of Gd_3Ni_4Y consisted of the two phases of $Gd_{1-x}Y_xNi$ (x=0.16) and $Gd_yY_{1-y}Ni$ (y=0.4). The X-ray diffraction analysis of the ternary samples near the composition near Gd_3Ni_4Y pointed to the absence of the ternary compound Gd_3Ni_4Y at 773 K. Similarly, it was found that the sample with composition $Gd_7Ni_{10}Y_3$ also contained the two phases of $Gd_{1-x}Y_xNi$ (x=0.16) and $Gd_yY_{1-y}Ni$ (y=0.4) pointing to the absence of compound $Gd_7Ni_{10}Y_3$ at 773 K. Fig. 8 presents the

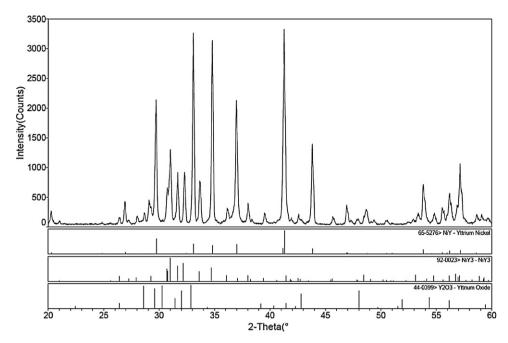


Fig. 6. The X-ray diffraction pattern of the sample no. C5 (Ni₄₀Y₆₀) containing the two phases of Y₃Ni and YNi. (The sample contains a little amount of Y₂O₃.).

X-ray diffraction pattern of the sample no. B15 ($Gd_{37.7}Ni_{50}Y_{12.3}$) with composition near $Gd_7Ni_{10}Y_3$ and Gd_3Ni_4Y consisted of the two phases of $Gd_{1-x}Y_xNi$ (x=0.16) and $Gd_yY_{1-y}Ni$ (y=0.4). No evidence was found to confirm the existence of the ternary compounds Gd_3Ni_4Y or $Gd_7Ni_{10}Y_3$ at 773 K. No new binary and ternary compound was found.

Six pairs of corresponding compounds in the Gd–Ni and Y–Ni systems, i.e., Y_2Ni_{17} and Gd_2Ni_{17} , $GdNi_5$ and YNi_5 , Gd_2Ni_7 and Y_2Ni_7 , $GdNi_3$ and YNi_3 , $GdNi_2$ and YNi_2 , Gd_3Ni and Y_3Ni were found to form a continuous series of solid solutions because these pairs of compounds have the same space groups and the same crystal structure, almost the same lattice parameters and similar characters [4–7], respectively. At the same time, metals Y and Gd can substitute for each other and form substitution solid solutions.

The solid solubility of Gd in the Y–Ni binary compounds and Y in Gd–Ni binary compounds were determined by X-ray diffraction technique using the phase disappearing method and lattice parameter method. By comparing the movement of the diffraction patterns of the single phases and the disappearance and the variations of the lattice parameters of the phases, the solid solubility of Gd or Y in these compounds were obtained. The variation of the lattice parameters of the compounds $Gd_yY_{1-y}Ni$ with the content of Gd from 0 to 25 at.% Gd (with y = 0 - 0.5) pointing to the solid solution $Gd_yY_{1-y}Ni$ existing with y = 0 - 0.4 (i.e. from 0 to 20 at.% Gd), as shown in Fig. 9. Since the variation of the lattice parameters on composition of $Gd_yY_{1-y}Ni$ (with y > 0.4) is different from those with y < 0.4, showing these samples with composition of $Gd_yY_{1-y}Ni$ (with y > 0.4) fell in the two-phase region of $Gd_{1-x}Y_xNi$

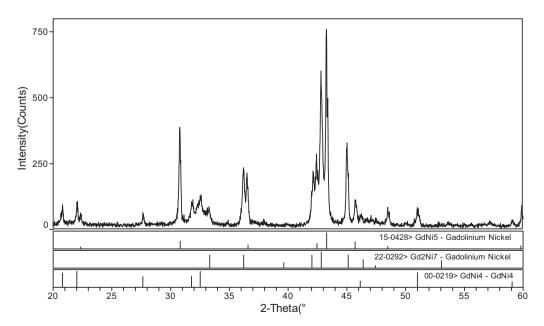


Fig. 7. The X-ray diffraction patterns of the sample no. A20 ($Gd_{20}Ni_{80}$).

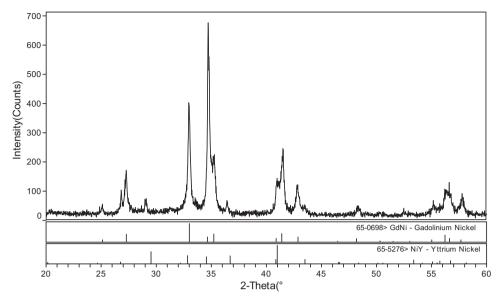


Fig. 8. The X-ray diffraction patterns of the sample no. B15 (Gd_{37.7}Ni₅₀Y_{12.3}).

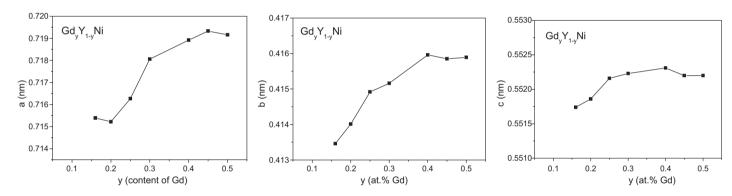


Fig. 9. The variation of the lattice parameters of the compounds $Gd_{\nu}Y_{1-\nu}Ni$ with the content of Gd.

 $(x=0.16)+Gd_yY_{1-y}Ni \ (y=0.4)$. This indicated that the maximum solid solubility of Gd in YNi is about 20 at.% Gd. Similarly, the maximum solid solubility of Y in GdNi were found to be about 8 at.% Y, respectively. No solubility of Gd in YNi₄ and Y in GdNi₄ was observed.

4. Conclusion

The isothermal section of the phase diagram of the Gd-Ni-Y ternary system at 773 K was investigated by means of X-ray powder diffraction, metallographic analysis and scanning electron microscopy with energy dispersive analysis. The isothermal section consists of 12 single-phase regions, 15 two-phase regions and 4 three-phase regions. Six pairs of corresponding compounds in the Gd-Ni and Y-Ni systems, i.e., Gd₂Ni₁₇ and Y₂Ni₁₇, GdNi₅ and YNi_5 , Gd_2Ni_7 and Y_2Ni_7 , $GdNi_3$ and YNi_3 , $GdNi_2$ and YNi_2 , Gd_3Ni and Y₃Ni and metals Gd and Y form a continuous series of solid solutions, respectively. At 773 K, the maximum solubilities of Gd in YNi and Y in GdNi were about 20 at.% Gd and 8 at.% Y, respectively. No solubility of Gd in YNi4 and Y in GdNi4 was observed. Under our experimental conditions, the binary compounds Gd₃Ni₂, Y₃Ni₂ and ternary compounds Gd₃Ni₄Y, Gd₇Ni₁₀Y₃ were not observed and no new binary or ternary compound was found in our binary and ternary alloy samples at 773 K.

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