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Phase diagram and compounds of the Gd–Fe–Nb ternary alloy system in the Fe-rich corner

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Abstract. The room-temperature phase diagram of the Gd–Fe–Nb ternary alloy system in the Fe-rich corner has been investigated by means of x-ray powder diffraction and thermomagnetic scans. In the system there exist $\text{Gd}_2\text{Fe}_{17-x}\text{Nb}_x$ ($x = 0\text{--}1.0$) '2:17' ($\text{Th}_2\text{Zn}_{17}$ -type structure), $\text{GdFe}_{11.6}\text{Nb}_{0.4}$ '1:12' (ThMn_{12} -type structure) and a new compound $\text{Gd}_3\text{Fe}_{29-x}\text{Nb}_x$ ($x = 0.9\text{--}1.3$, Curie temperature $T_C = 236\text{--}243^\circ\text{C}$). The homogeneous ranges of the three above mentioned compounds are determined by a precise lattice parameter method. Further annealing the new compounds $\text{Gd}_3(\text{Fe}, \text{Nb})_{29}$ at 700°C for 10 days results in no trace of decomposition, implying that $\text{Gd}_3(\text{Fe}, \text{Nb})_{29}$ may be a room-temperature phase.

1. Introduction

The past decade has witnessed a renewed interest in the structural, magnetic properties and phase relations of rare-earth–iron intermetallic compounds. $\text{Nd}_2\text{Fe}_{14}\text{B}$ has many excellent properties for permanent magnet materials, but its low Curie temperature (310°C) limits its applications above 150°C . Before 1992, most of the researchers focused on two families of intermetallics, the rhombohedral $\text{R}_2(\text{Fe}, \text{M})_{17}$ ('2:17' phase, $\text{Th}_2\text{Zn}_{17}$ type) and $\text{R}(\text{Fe}, \text{M})_{12}$ ('1:12' phase, ThMn_{12} type), where R is a rare-earth element and M is an element for stabilizing the phases. Recently a new class of compounds, monoclinic $\text{R}_3(\text{Fe}, \text{M})_{29}$ [1] ('3:29' phase, $\text{Nd}_3(\text{Fe}, \text{Ti})_{29}$ type) attracted special attention because of their novel structure and particular magnetic properties. All the '2:17', '3:29' and '1:12' structures can be derived from the RT_5 (CaCu_5 type) structure by substitution of R for different T–T (T: transition element) dumbbell numbers, the crystallographic relationship between the three major compounds mentioned above and RT_5 were discussed in detail by Li *et al* [2, 3]. The stabilizing elements M are needed to form '3:29' compounds. Usually, the larger the atomic radius of M, the lower the content of M needed [4–8]. Except $\text{M} = \text{Ti}$, $\text{V}(\text{V} + \text{Al})$, Cr , Mn and Mo , other M substitution for Fe to form '3:29' phase have not been reported. Generally speaking, the third nonmagnetic element M has a detrimental influence on both the Curie temperature (T_C) and spontaneous magnetization. Thus, for the purpose of application the smallest possible content of M is preferred. Considering

Nb's larger radius and the existence of the compound $R(\text{Fe}, \text{Nb})_{12}$ [9, 10], we surmized the possibility of synthesizing a new '3:29' compound at lower Nb content. In this paper, we present a systematic investigation of the room temperature phase diagram in the Fe-rich corner ($\text{Nb} < 35 \text{ at.}\%$, $\text{Gd} < 35 \text{ at.}\%$) in the ternary system Gd–Fe–Nb.

2. Experimental procedure

64 samples of alloys with various compositions weighing about 4 g were prepared from Gd, Fe and Nb of $>99.9\%$ purity by argon arc-melting on a water-cooled copper hearth. The weight losses of each sample are lower than 1%. The samples were then wrapped in tantalum foil, encapsulated in sealed vacuum quartz tubes and annealed for 2 weeks at 1000°C , and then slowly cooled to room temperature in the following three days. Chemical analyses were performed for some pure phases to obtain their exact compositions. Two of the pure '3:29' samples were given an additional annealing treatment at 700°C for 10 days. Phase identifications were made on a Guinier–de Wolff camera with $\text{Co } K\alpha$ radiation and thermomagnetic scans at low magnetic fields (0.04 T). For determination of the lattice constants, the diffraction data were collected on a Philips X'pert diffractometer with $\text{Cu } K\alpha$ radiation with graphite monochromator. The data were collected using step scan mode with a scanning step of 0.02° and a sampling time of 5 seconds, 2θ from 10° to 90° . The NBS SRM. 640 Si was added as internal standard to calibrate the 2θ position.

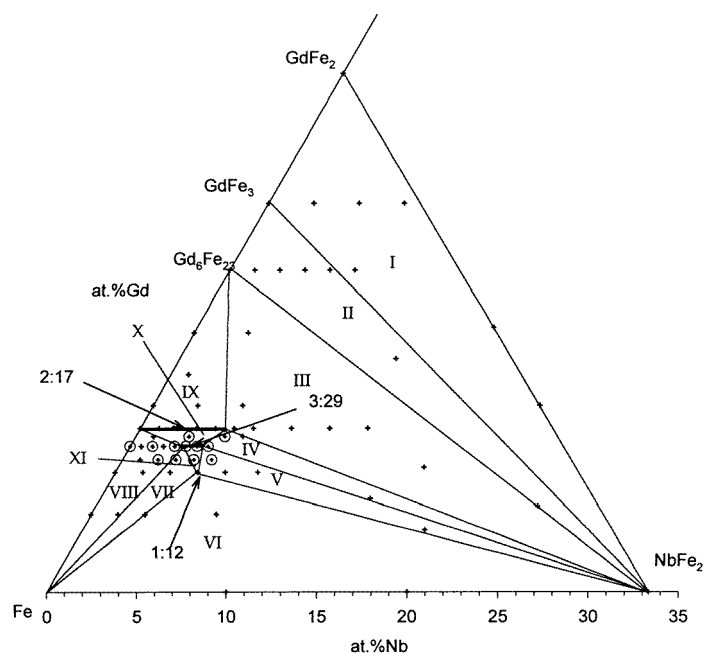


Figure 1. The phase diagram of the Gd–Fe–Nb ternary system in the Fe-rich region at room temperature. '+' represents results obtained by x-ray diffraction phase identification, and 'O' represents results obtained by thermomagnetic scans (see figure 2). I. $\text{GdFe}_2 + \text{GdFe}_3 + \text{NbFe}_2$, II. $\text{GdFe}_3 + \text{Gd}_6\text{Fe}_{23} + \text{NbFe}_2$, III. '2:17' + $\text{Gd}_6\text{Fe}_{23} + \text{NbFe}_2$, IV. '2:17' + '3:29' + NbFe_2 , V. '3:29' + '1:12' + NbFe_2 , VI. '1:12' + $\alpha\text{-Fe} + \text{NbFe}_2$, VII. '1:12' + '3:29' + $\alpha\text{-Fe}$, VIII. '3:29' + '2:17' + $\alpha\text{-Fe}$, IX. $\text{Gd}_6\text{Fe}_{23} + \text{'2:17'}$, X. '2:17' + '3:29', XI. '3:29' + '1:12'.

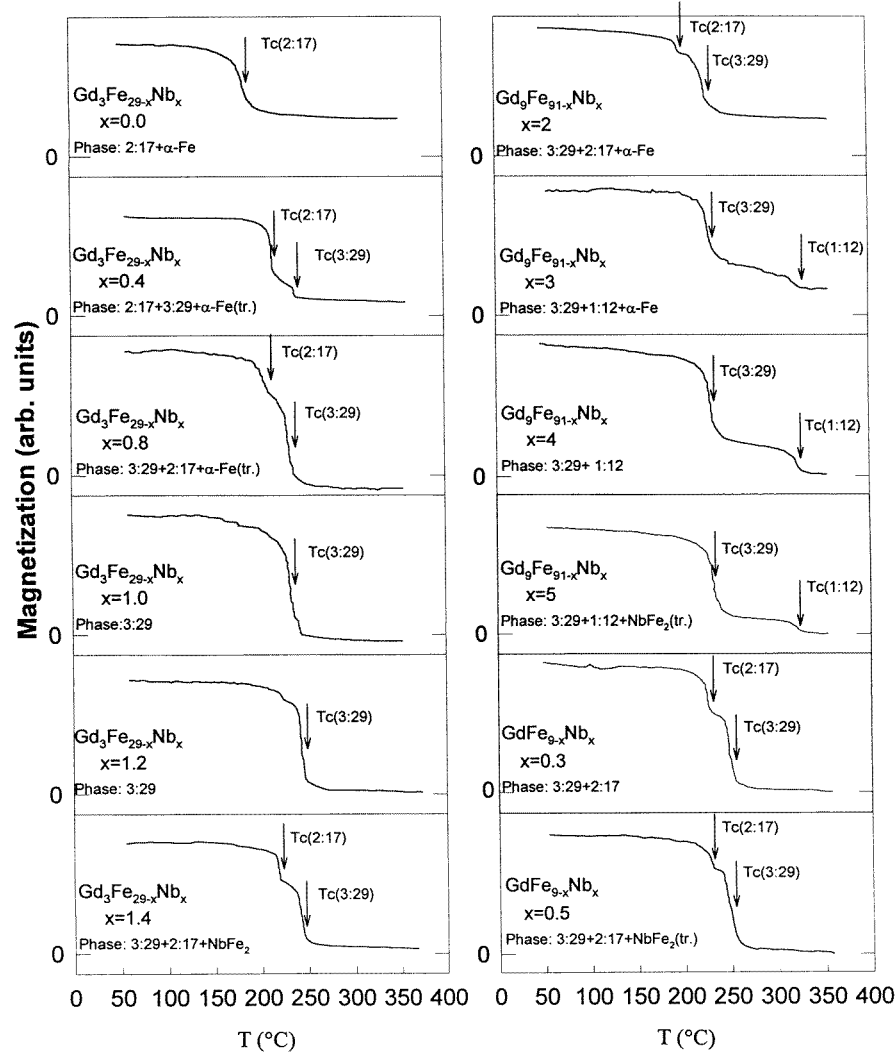


Figure 2. Thermomagnetic curves of samples (marked with ‘O’ in figure 1) near ‘3:29’ phase (magnetic field: 0.04 T). The Curie temperatures (T_C) of ‘2:17’, ‘3:29’ and ‘1:12’ compounds are marked by the arrows.

3. Results and discussion

Detailed analysis of the x-ray diffraction patterns of samples in the Fe-rich corner revealed the existence of two ternary compounds, $\text{Gd}(\text{Fe}, \text{Nb})_{12}$, $\text{Gd}_3(\text{Fe}, \text{Nb})_{29}$ and a solid solution $\text{Gd}_2(\text{Fe}, \text{Nb})_{17}$. The compound $\text{Gd}_3(\text{Fe}, \text{Nb})_{29}$ was first identified in this study. The phase diagram can be divided into eight three-phase and three two-phase regions (figure 1). Since the structure of ‘3:29’ compound is intermediate between the rhombohedral ‘2:17’ and tetragonal ‘1:12’ structures, their similar diffraction patterns make it difficult to determine the phase relationship near the ‘3:29’ compound. The different Curie temperatures of these compounds help us distinguish these phases from each other. 12 samples near the ‘3:29’

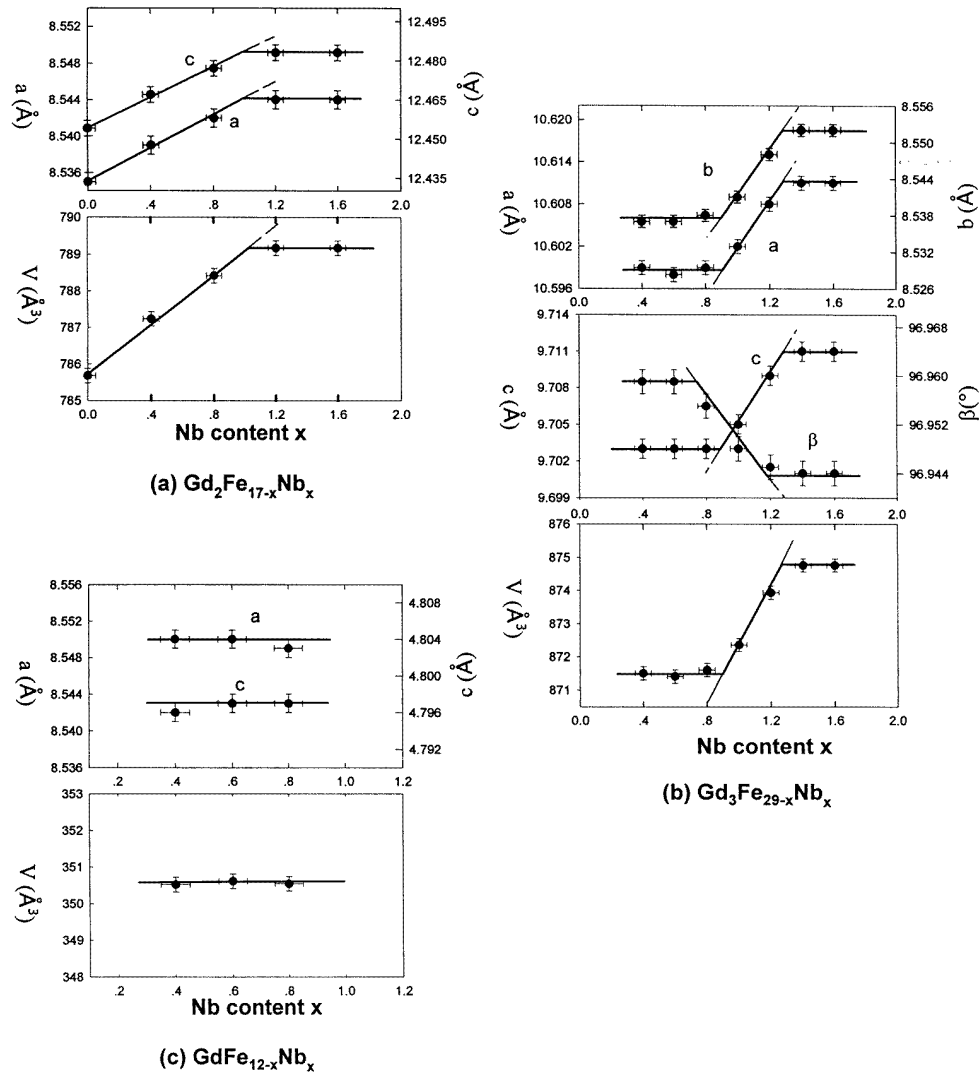


Figure 3. Dependence of the lattice parameters of three compounds on the Nb content x ; the error bars show the error ranges of compositions and lattice parameters. (a) $Gd_2Fe_{17-x}Nb_x$, (b) $Gd_3Fe_{29-x}Nb_x$, (c) $GdFe_{12-x}Nb_x$.

compound were selected to perform thermomagnetic scans. The thermomagnetic curves of these samples are shown in figure 2. The existence of '2:17', '3:29' and '1:12' compounds is evidenced by T_C values in the ranges 192–231, 236–243 and 320 °C respectively; $NbFe_2$ and α -Fe are ruled out since their T_C values are -73 °C and 770 °C respectively. Figure 3 shows the compositional dependence of the lattice parameters of the '2:17', '3:29' and '1:12' compounds. Linear increases of cell volume of $Gd_2Fe_{17-x}Nb_x$ and $Gd_3Fe_{29-x}Nb_x$ with the Nb content x are observed; indicating that the homogeneous ranges of '2:17' and '3:29' are $0 \leq x \leq 1.0$ (Nb/Fe = 0–6.3 at.%) (figure 3(a)) and $0.9 \leq x \leq 1.3$ (Nb/Fe = 3.2–4.7 at.%) (figure 3(b)) respectively. There is no obvious change in the cell volume of $GdFe_{12-x}Nb_x$ within the error range for the composition of alloys near the pure phase $GdFe_{11.4}Nb_{0.6}$

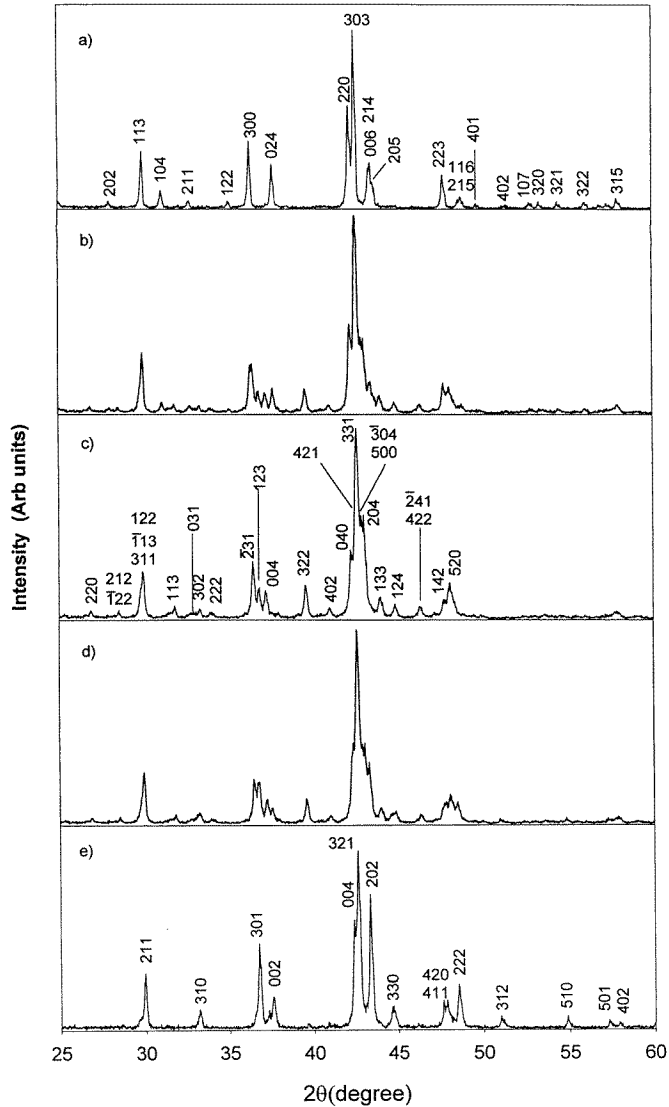


Figure 4. X-ray diffraction patterns using Cu $K\alpha$ radiation for the samples of the Fe-rich Gd-Fe-Nb system: (a) $Gd_2Fe_{16.2}Nb_{0.8}$ ('2:17' pure phase), (b) $GdFe_{8.7}Nb_{0.3}$ (two phase: '2:17' + '3:29'), (c) $Gd_3Fe_{28}Nb$ ('3:29' pure phase), (d) $Gd_9Fe_{86}Nb_5$ (two phase: '1:12' + '3:29'), (e) $GdFe_{11.4}Nb_{0.6}$ ('1:12' pure phase).

($0.4 \leq x \leq 0.8$) (figure 3(c)). According to the Gibbs phase rule, this means that its homogeneous range must be very narrow. All the data of homogeneous regions and Curie temperatures of the three above mentioned compounds are listed in table 1.

X-ray diffraction patterns of the three pure phases and two samples containing two phases are shown in figure 4. The '1:12' phase (figure 4(e)) has the tetragonal $ThMn_{12}$ -type structure, the '2:17' phase (figure 4(a)) has the rhombohedral Th_2Zn_{17} type and $Gd_3(Fe, Nb)_{29}$ (figure 4(c)) is isostructural with $Nd_3(Fe, Ti)_{29}$, with monoclinic system ($P2_1/c$) [7, 8, 11, 12]. Rietveld powder analysis was performed to refine the structure of

Table 1. Homogeneous regions and Curie temperatures of the three major compounds. T_C is the Curie temperature (in °C).

Compounds	Crystal type	Nb/Fe (at.%)	T_C (°C)
Gd ₂ (Fe, Nb) ₁₇	Th ₂ Zn ₁₇ type	0–6.3	192–231
Gd ₃ (Fe, Nb) ₂₉	Nd ₃ (Fe, Ti) ₂₉ type	3.2–4.7	236–243
Gd(Fe, Nb) ₁₂	ThMn ₁₂ type	5.3	320

Gd₃Fe₂₈Nb. The lattice parameters are: $a = 10.601(2)$ Å, $b = 8.541(1)$ Å, $c = 9.704(1)$ Å and $\beta = 96.948(2)^\circ$ ($R_p = 8.98\%$, $R_{wp} = 12.02\%$, $R_{exp} = 6.43\%$). A significant difference of Gd₃(Fe, Nb)₂₉ from other ‘3:29’ compounds is that it is stable at room temperature, while other ‘3:29’ compounds only exist in a much higher temperature region ($\sim 1100^\circ\text{C}$) [13]. Further annealing the compounds at a temperature of 700°C for 10 days, we did not find any trace of other phases, both by x-ray analysis and thermal-magnetic scans.

4. Conclusion

We have constructed a room temperature equilibrium-phase diagram of the Gd–Fe–Nb ternary system in the compositional region Nb < 35 at.%, Gd < 35 at.% for the Fe-rich corner, and determined the lattice parameters and Curie temperature of Gd₂(Fe, Nb)₁₇, tetragonal Gd(Fe, Nb)₁₂ and monoclinic Gd₃(Fe, Nb)₂₉. The compound Gd₃(Fe, Nb)₂₉ was first identified in this study. Unlike other ‘3:29’ compounds, it is a stable phase at room temperature.

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