

PHASE EQUILIBRIA AMONG γ , Ni_3Nb - δ AND Fe_2Nb - ϵ PHASES IN Ni-Nb-Fe AND Ni-Nb-Fe-Cr SYSTEMS AT ELEVATED TEMPERATURES

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Abstract

Phase equilibria in Ni-Nb-Fe ternary and Ni-Nb-Fe-Cr quaternary systems at elevated temperatures have been examined. In the ternary system, Ni_3Nb - δ (D0_a) single phase field expands along the equi-niobium concentration up to 10% Fe at 1473 K. The δ phase becomes in equilibrium with $(\text{Fe}, \text{Ni})_2\text{Nb}$ - ϵ (C14 Laves) phase with a composition of Ni-30Fe-26Nb and γ phase with its composition of Ni-35Fe-10Nb. A new intermetallic phase is found at compositions around Ni-22Nb-20Fe, and the crystal structure is identified to be ordered hexagonal (hP24 with stacking sequence of abcbcb). This phase is in equilibrium with γ and δ phases, and no δ + γ + ϵ three-phase coexisting region exists in this system. In Fe-20Cr-(25-40)Ni-(2-6)Nb quaternary system, four intermetallic phases of δ , C14 Laves (ϵ), C15 Laves (Cr_2Nb) and $(\text{Ni}, \text{Fe})_6\text{Nb}_7$ - μ phases are found to exist in γ matrix, and the isothermal tetrahedron at 1473 K in this system is constructed. The γ -Ni solid solution phase becomes in equilibrium with C14 Laves phase in Ni poor side (less than 30%), whereas it is in equilibrium with C15 Laves phase in Ni rich side (~40%). At the very limited region between them, there is a small window opened where the γ phase can be in equilibrium with μ phase. However, at 1073 K, only δ and C14 phases become in equilibrium with γ phase. The changes in the phase equilibria with temperature is caused by the occurrence of two transition peritectoid-type reactions: (I) $\gamma + \text{C15} \rightarrow \delta + \mu$, (II) $\gamma + \mu \rightarrow \delta + \text{C14}$.

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Introduction

The intermetallic compound Ni_3Nb (δ) is of interest as a strengthener of Ni base alloys because of its phase stability up to the melting temperature [1-3]. The crystal structure of the δ phase is ordered orthorhombic (D0_a structure), the lowest crystal symmetry among the A_3B compounds with close-packed structure [4, 5]. Thus, due to its low crystal symmetry the δ phase precipitates in γ -Ni matrix with Widmannstätten morphology, with a crystallographic orientation relationship of $\{111\}_\gamma // (010)_\delta$, $\langle 1\bar{1}0 \rangle_\gamma // [100]_\delta$ [6]. The addition of Fe to Ni-Nb binary alloys is known to promote the formation of metastable $\text{Ni}_3\text{Nb}-\gamma'$ phase with D0_{22} structure at relatively low temperatures [7], although the detail mechanism of the formation has not yet been fully understood. Recently, Takeyama et al. [8] has revealed that Fe addition has another effect to refine the δ precipitates drastically, and they attributed the Fe effect to the lattice misfit change between the two phases, specifically along the preferred growth direction. Thus, Fe plays an important role in changing not only the type of precipitates but also its morphology of Ni-Nb based alloys. In fact, commercial alloys strengthened by $\text{Ni}_3\text{Nb}-\gamma'$ phase, such as Alloy 718 and 706, contains a large amount of Fe. Therefore, understanding the role of Fe in the microstructure evolution is a key for alloy development applicable at elevated temperatures.

There are a number of reports on the ternary phase diagram study of Ni-Fe-Cr, Ni-Nb-Cr and Fe-Nb-Cr systems at elevated temperatures [9]. However, as far as the authors are aware, no comprehensive study on the phase diagram of Ni-Nb-Fe ternary system has been reported. If the phase diagram of this ternary system is established, and combined these four ternary systems together, phase equilibria in the quaternary system of Ni-Nb-Fe-Cr can be somehow understood. Recently we have studied phase equilibria in several Ni-Nb-M (Fe, Co, Al, Ti) ternary systems at elevated temperatures [10-12], but these studies are limited to the solid solution field around $\text{Ni}_3\text{Nb}-\delta$ phase.

In this study, we first examine the phase equilibria in Ni-Nb-Fe ternary system at elevated temperatures. Then, by fixing the Cr content of 20 at % and changing the content of Fe/Ni or Fe/Nb ratio, phase equilibria as well as microstructure evolution in Ni-Nb-Fe-20Cr quaternary system have been studied, particularly paying attention to the intermetallic compounds formed in this system.

Experimental

The ternary Ni-Nb-Fe alloys used in this study have composition ranges of Ni-(15-40) at % Nb-(0-15) at % Fe. In addition, six quaternary alloys with compositions having different Fe/Ni or Fe/Nb ratio of a base alloy Fe-20Cr-40Ni-2Nb were used. (all compositions are hereafter given in at % unless otherwise stated). They were prepared by argon arc-melting with non-consumable tungsten electrode as 30 g button ingots, using high purity metals. Each button ingot was melted six times by turning over each time in order to avoid segregation. For phase diagram study, these alloys were first homogenized at 1523 K/ 24 h, and then equilibrated at 1473 K/ 240 h. The binary Ni-40.5 Nb alloy was unidirectionally solidified (UDS) by optical floating zone furnace. For study on microstructure evolution, some of the quaternary alloys were aged at 1073 K for a certain period of time. The heat treatment samples with a size of 6 x 6 x 10 mm were cut by electro-discharged machining, and they were sealed off in silica

capsules under argon back-filled after evacuating to 8×10^{-4} Pa. Microstructures were examined by scanning electron microscope (SEM) and transmission electron microscope (TEM). SEM samples were mechanically polished, followed by electropolishing in a solution of phosphoric acid with supersaturated chromic anhydride at a potential of 10 V at 373 K. The chemical compositions of the phases present in the equilibrated samples were analyzed by an electron probe microanalyzer (EPMA) equipped with a wavelength dispersive spectrometer under an operating condition of 20 kV/ 2×10^{-8} A. The phase determination was done by powder X-ray diffraction (XRD) and TEM. TEM foils were prepared by twin-jet electropolishing in a electrolyte of ethanol with 12% perchloric acid at 253 K.

Results and discussion

Phase equilibria in Ni-Nb-Fe ternary system

Backscattered electron images (BEIs) of some of the alloys are shown in Fig. 1, and the phases present and their analyzed compositions of all the alloys equilibrated at 1473K are summarized in Table 1. A series of alloys with 15 Nb shows basically two phases of γ and δ . In Ni-15Nb-10Fe, the microstructure exhibits granular morphology, and the bright and dark phases are γ and δ , respectively (Fig. 1(a)). In case of Ni-15Nb-25Fe (Fig. 1(b)), however, although the dark one is γ matrix phase, the bright phase is characterized into two types in shape: one bar-like and the other large spherical morphology. The former is identified to be δ with its

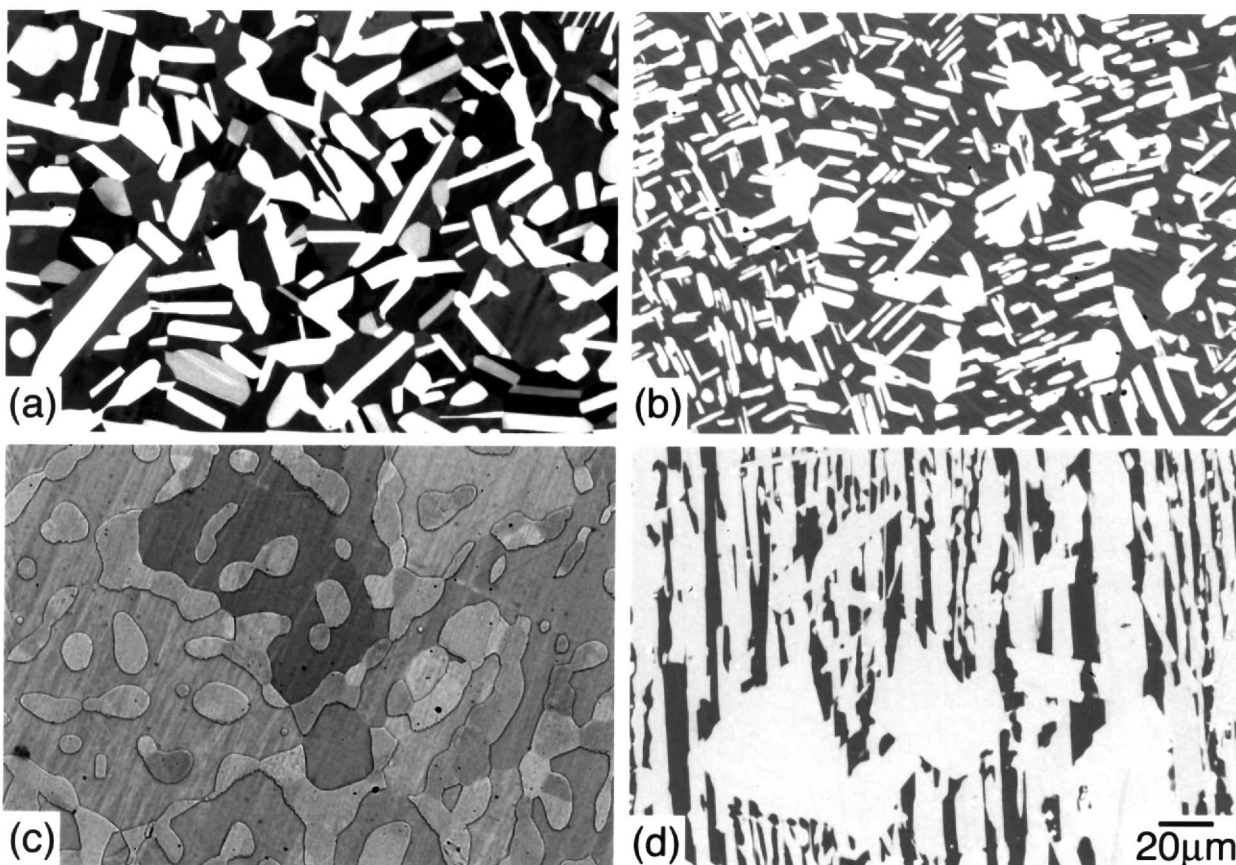


Fig. 1 BEIs of the alloys equilibrated at 1473K for 240h : (a) Ni-15Nb-10Fe, (b) Ni-15Nb-25Fe, (c) Ni-25Nb-15Fe and BEI of (d) Ni-40.5Nb UDS alloy.

composition of Ni-23.1Nb-10.3Fe, whereas the latter is apparently a different phase with its Fe content nearly twice as high as that of the δ . The crystal structure of this unknown phase is discussed later in this section. The alloy Ni-25Nb-15Fe shows dual phase microstructure (Fig. 1(c)). The matrix phase is δ , and it is in equilibrium with (Fe, Ni)₂Nb- ϵ Laves phase with hexagonal C14 structure. The UDS Ni-40.5 Nb alloy consists of δ and Ni₆Nb₇- μ phases, and due to an eutectic reaction of L \rightarrow δ + μ , the microstructure exhibits a lamellar morphology with some primary phase of μ (Fig. 1(d)), as expected from the published phase diagram [13]. However, it should be noted that the composition homogeneity region from the stoichiometry in Nb-rich side is very limited to 0.2% of the δ phase, unlike the published data [13].

Table 1 EPMA analysis of the phases present in Ni-Nb-Fe ternary alloys equilibrated at 1473K.

Bulk alloy compositions	Phases present	Analyzed composition (at.%)		
		Ni	Nb	Fe
Ni-16Nb	γ	87.8	12.2	-
	δ	76.6	23.4	-
Ni-15Nb-5Fe	γ	81.9	11.2	6.9
	δ	75.8	22.1	2.1
Ni-15Nb-10Fe	γ	75.6	9.8	14.6
	δ	73.2	23.4	3.4
Ni-15Nb-15Fe	γ	68.2	8.7	23.1
	δ	72.6	22.7	4.7
Ni-15Nb-25Fe	γ	55.1	9.3	35.6
	δ	66.6	23.1	10.3
	unknown	57.7	21.9	20.4
Ni-25Nb-5Fe	δ	70.4	24.4	5.1
Ni-25Nb-15Fe	δ	67.2	24.3	8.5
	ϵ	44.5	26.1	29.4
Ni-40.5Nb	δ	74.8	25.2	-
	μ	52.5	47.5	-

Figure 2 shows an isothermal section of Ni-rich corner of Ni-Nb-Fe ternary system at 1473 K. The δ single phase field expands toward the equi-niobium concentration direction up to about 10 at%, and beyond that, the δ phase becomes in equilibrium with ϵ -(Fe, Ni)₂Nb Laves phase. In γ + δ two-phase region, the Nb concentration of γ and δ phases in equilibrium each other is 12.2 at % and 23.4 at%, respectively, in binary case, consistent with the published data [13]. In ternary case, the Nb concentration of δ phase in equilibrium with γ phase remains almost unchanged, regardless of Fe content. However, Fe atoms tends to partition to γ phase, and the tendency becomes more obvious with increasing Fe content. Consequently, the δ phase can be in equilibrium with γ phase with its composition up to Ni-36Fe-9Nb. It should be noted that there exists no δ + γ + ϵ three-phase tie triangle, and within this region the unknown phase exists at the composition of Ni-22Nb-20Fe, as mentioned in Fig. 1(b). In addition, we confirmed that the alloy with composition of Ni-20Nb-25Fe is completely melted at 1473 K, indicating an existence of liquid phase region within the δ + γ + ϵ triangle.

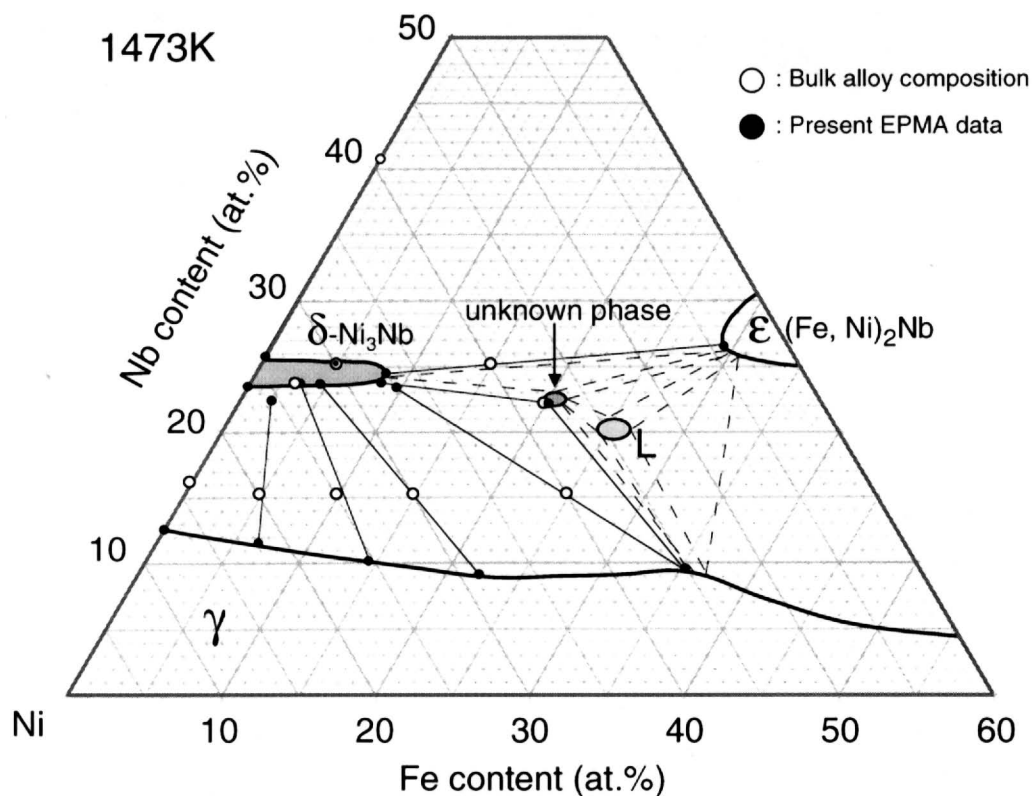


Fig. 2 Isothermal section of Ni-Nb-Fe ternary system at 1473K.

Structure of the unknown phase

In order to identify the crystal structure of the unknown phase observed in Fig. 1(b), the alloy with its analyzed composition in Table 1 is prepared by arc melting. Figure 3 shows a BEI of the alloy heat treated at 1473 K/240 h. The microstructure becomes almost a single phase, although a small amount of liquid phase is formed along the grain boundaries. Figure 4 shows selected area diffraction patterns (SADPs) of TEM taken from the sample with various incident

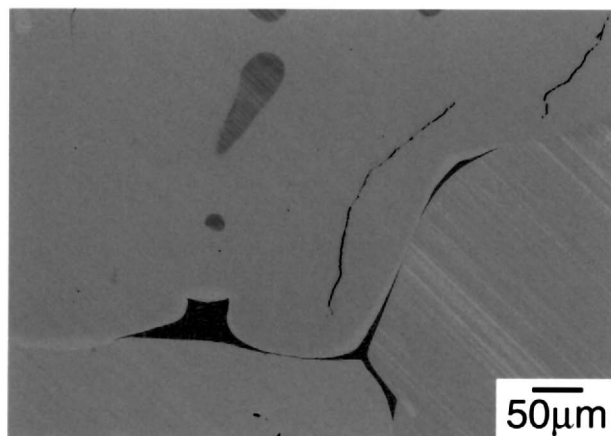


Fig. 3 BEI of Ni-22Nb-20Fe heat treated at 1473K / 240h.

beam directions, **B**. Fig. 4 (a) clearly shows the six-fold symmetry pattern, indicating hexagonal structure. The superlattice reflections are observed just in half of both fundamental reflections $\mathbf{g}_1 = 11\bar{2}0$ and $\mathbf{g}_2 = 10\bar{1}0$ (\mathbf{g} : reciprocal lattice vector). Figs. 4 (b) and (c) represent the patterns obtained from $\mathbf{B} = 11\bar{2}0$ and $10\bar{1}0$, respectively, normal to that $\mathbf{B} = 0001$ in Fig. 4(a). In both patterns, there are 5 superlattice spots along the fundamental reflections of $\mathbf{g}_3 = 0001$. From these results, the structure of the unknown phase is A_3B type ordered hexagonal structure of hP24, where the arrangement of *b*-sublattice on the basal plane becomes triangular array (T-type) and its stacking sequence is abcbcb. Liu et al. [14] reported that a partial replacement of Ni with Fe and Co in Ni_3V changes its crystal structure more symmetrical from D0_{22} (R-type: rectangular array of *b*-sublattice on the close-packed plane) to L1_2 (T-type). As shown in Fig. 2, the δ solid solution field extends toward the equi-niobium concentration direction, indicating that Fe atoms preferentially occupy *a*-sublattice (Ni) site. Thus, just like the case of Ni_3V , the poor symmetrical structure of D0_a in Ni_3Nb becomes more symmetrical by the addition of Fe. The details of the structure analysis as well as the structural change will be discussed elsewhere [15].

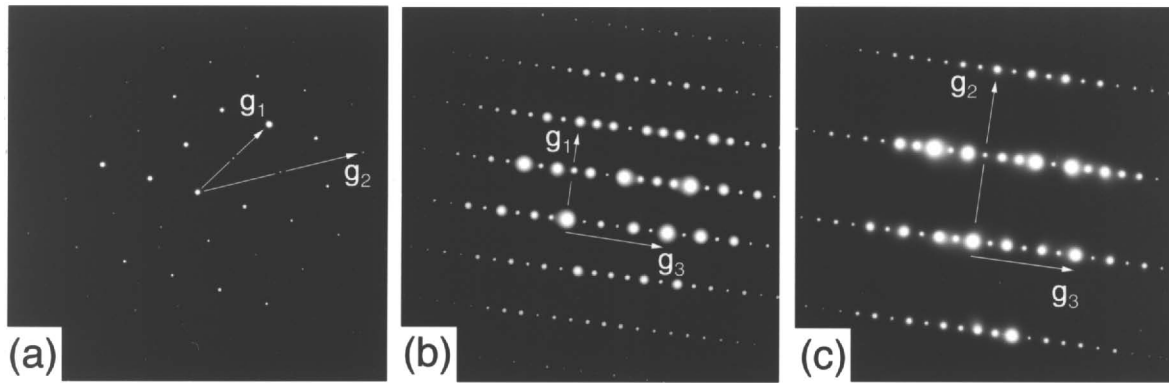


Fig. 4 SADPs obtained from the unknown phase in Fig. 3 : (a) $\mathbf{B} = [0001]$, (b) $\mathbf{B} = [11\bar{2}0]$, (c) $\mathbf{B} = [10\bar{1}0]$

Phase equilibria in Ni-Nb-Fe-Cr quaternary system

BEIs of the quaternary alloys equilibrated at 1473 K/240 h are shown in Fig. 5. The base alloy of Fe-20Cr-40Ni-2Nb exhibits γ single phase microstructure (Fig. 5(a)). The single phase microstructure is obtained even though Ni content is decreased to 30%, but if decreased to 25%, the second phase with brighter contrast appears sparsely in the matrix, as shown in Figs. 5(b) and (c). On the other hand, when increasing Nb content of the base alloy to 4%, small particles are formed. Table 2 summarizes the phases present and their analyzed compositions of the alloys with second phase. The bright phase in Fig. 5(c) is rich in Nb and identified as C14 Laves (Fe_2Nb - ϵ) phase. The second phase in Fig. 5(d) contains a huge amount of Nb of 46 %, and this phase is identified as $(\text{Ni}, \text{Fe})_6\text{Nb}_7$ - μ phase. It should be noted here that when Nb content of the base alloy is increased to 6%, a second phase different from μ phase appears, and it is identified as C15 Laves (Cr_2Nb type) phase. As shown in Table 2, its Nb concentration is reduced to 25% and, instead, Ni content is rather higher.

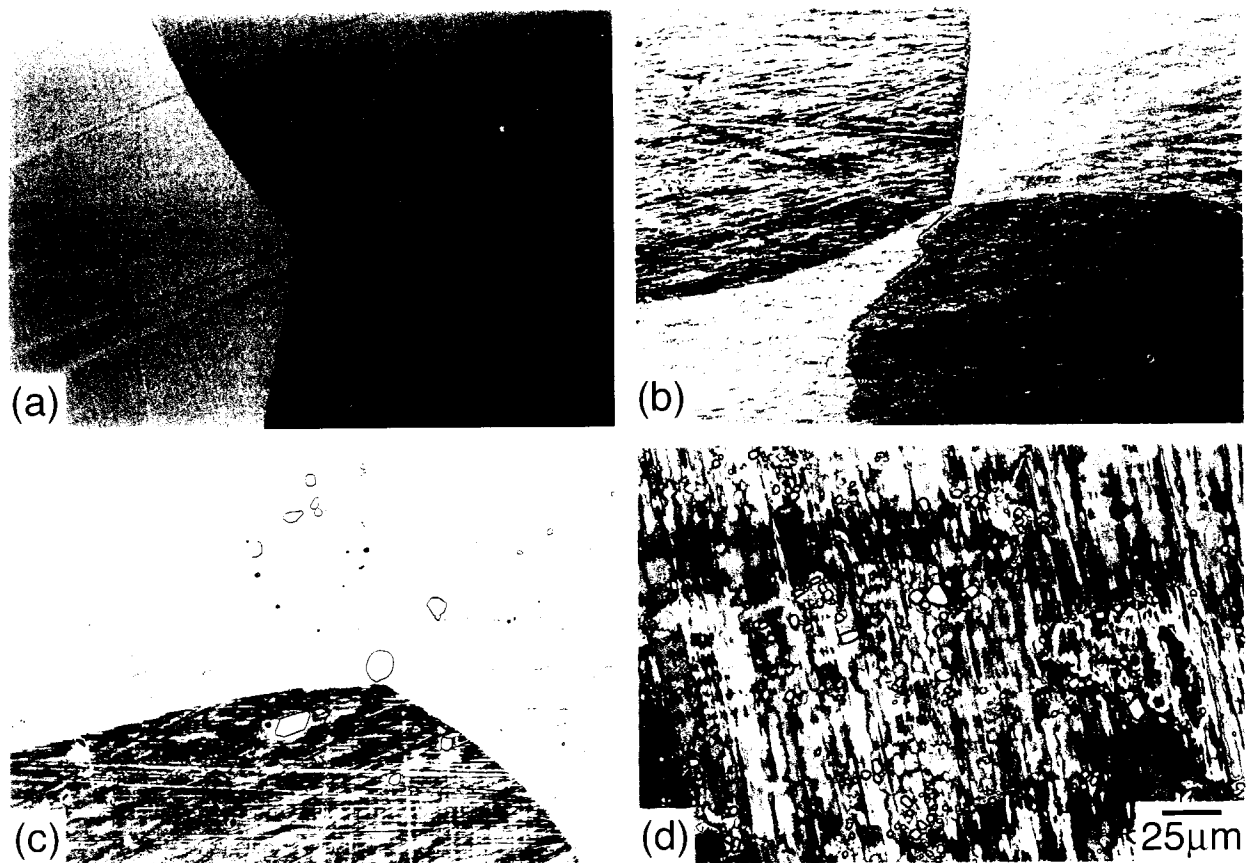


Fig. 5 BEIs of the quaternary alloys equilibrated at 1473K for 240h : (a) Fe-20Cr-40Ni-2Nb, (b) Fe-20Cr-30Ni-2Nb, (c) Fe-20Cr-25Ni-2Nb, (d) Fe-20Cr-40Ni-4Nb.

Table 2 EPMA analysis of the phases present in Ni-Nb-Fe-Cr quaternary alloys equilibrated at 1473K.

Bulk alloy compositions	Phases present	Analyzed composition (at.%)			
		Fe	Cr	Ni	Nb
Fe-20Cr-25Ni-2Nb	γ	53.1	20.7	24.3	1.9
	C14(Fe_2Nb)	42.8	13.7	17.8	25.7
Fe-20Cr-40Ni-4Nb	γ	36.4	20.6	39.3	3.7
	μ	20.2	13.3	20.6	45.9
Fe-20Cr-40Ni-6Nb	γ	35.0	21.1	39.8	4.1
	C15(Cr_2Nb)	28.5	14.5	32.1	24.9

Based on these results, together with the published ternary phase diagram, the isothermal tetrahedron at 1473 K in Fe-Cr-Ni-Nb quaternary system is constructed, which is illustrated in Fig. 6. Among the alloys studied, the γ -Ni solid solution phase becomes in equilibrium with C14 Laves phase in Ni poor side (less than 30%), whereas it is in equilibrium with C15 Laves phase in Ni rich side (~40%). At the very limited region between them, there is a small window opened where the γ phase can be in equilibrium with μ phase.

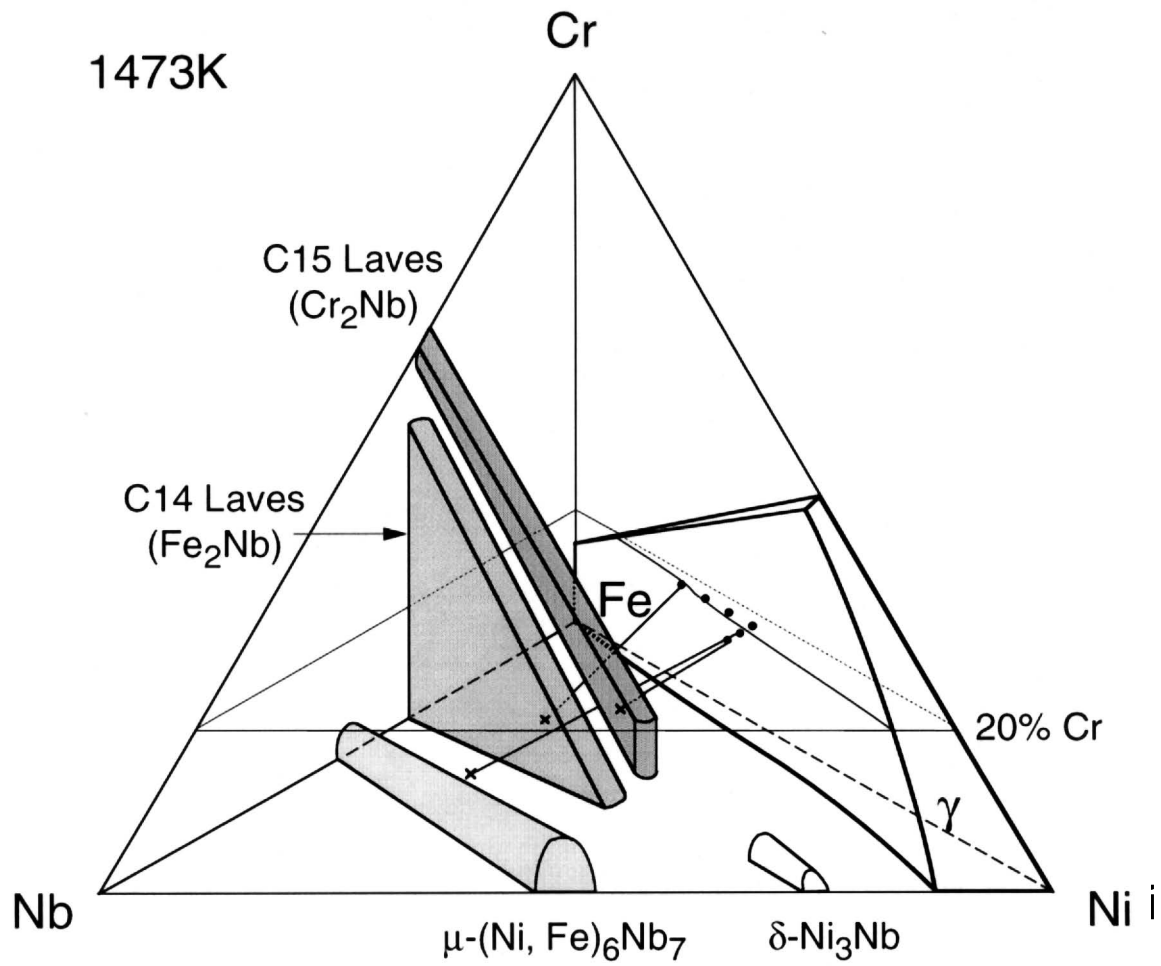


Fig. 6 The isothermal tetrahedron at 1473K in Ni-Nb-Fe-Cr quaternary system, showing intermetallic compounds in equilibrium with γ -Ni solid solution.

Microstructure evolution in the quaternary system

The quaternary alloys equilibrated at 1473 K were subsequently aged at 1073 K, and the microstructures were examined. Figure 7 shows BEIs of the alloys corresponding to Fig. 5. The base alloy exhibits precipitates with Widmannstätten morphology, and because of the morphology, these precipitates appear to be Ni_3Nb - δ phase. In the alloys with lower Ni content, very fine spherical particles are homogeneously precipitated in the γ matrix, as shown in Figs. 7(b) and (c). The precipitated phase in both alloys is C14 Laves phase. We confirmed that the alloy with 35% Ni exhibits both δ and C14 phases, although it is not shown here. Thus, it is found that at 1073 K the type of precipitate changes from δ to C14 with decreasing Ni/Fe ratio in Fe-20Cr-40Ni-2Nb alloy. On the other hand, the alloy with high Nb shows Widmannstätten microstructure, together with some lamellar structure (Fig. 7(d)). Note that the Widmannstätten morphology is much finer than that in the base alloy (Fig. 7(a)). The similar microstructure was also observed in the alloy with 6% Nb, although the lamellar morphology is more obvious and dominant. These precipitates are confirmed to be Ni_3Nb - δ phase. Ghanem

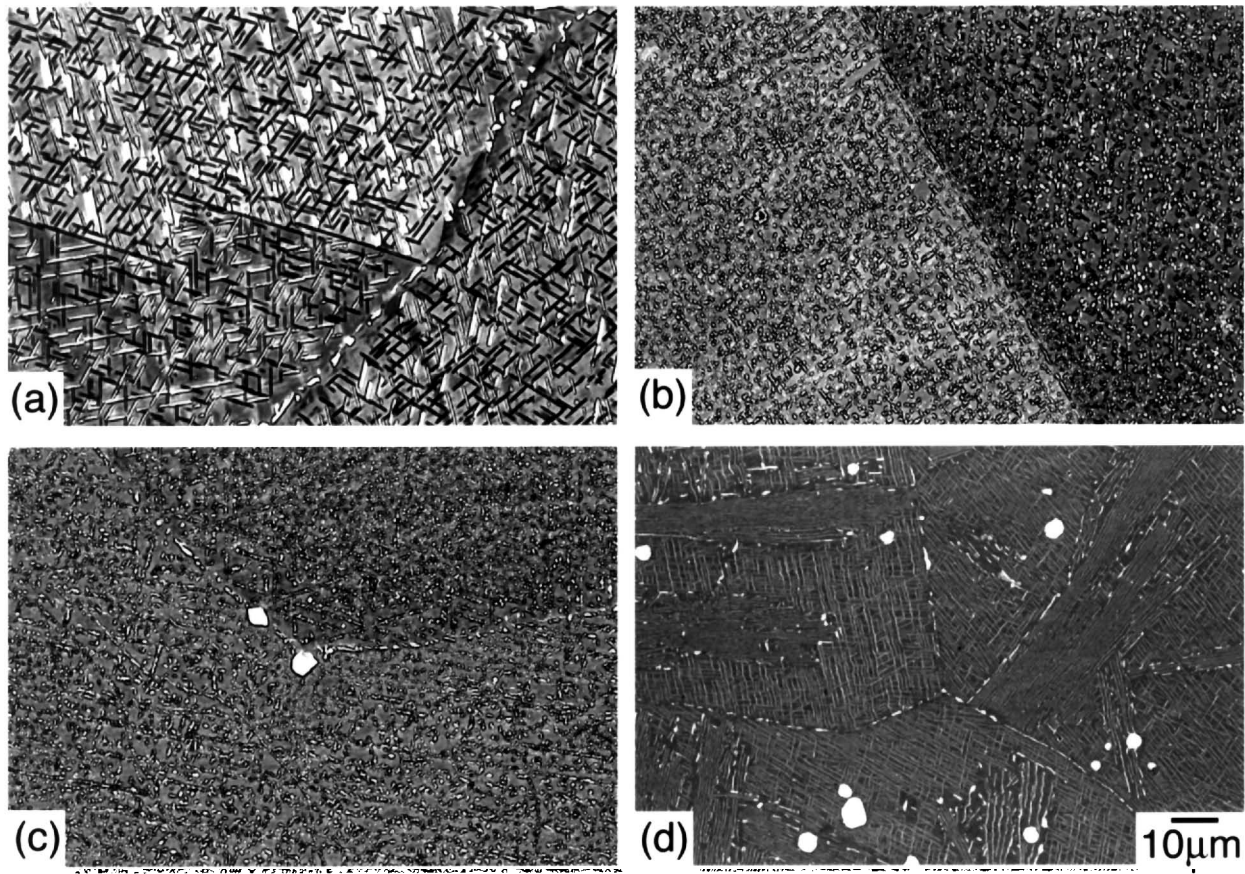
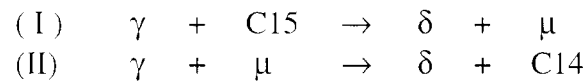


Fig. 7 BEIs of the alloys aged at 1073K for 240h : (a) Fe-20Cr-40Ni-2Nb, (b) Fe-20Cr-30Ni-2Nb, (c) Fe-20Cr-25Ni-2Nb, (d) Fe-20Cr-40Ni-4Nb.

[11] reported that in Ni-12Nb-5Fe the metastable $\text{Ni}_3\text{Nb}-\gamma'$ phase precipitated in the γ matrix in the early stage of aging at 1073 K changes to stable $\text{Ni}_3\text{Nb}-\delta$ phase through discontinuous precipitation, resulting in γ/δ lamellar morphology. Recently we also recognized such morphology change of $\text{Ni}_3\text{Nb}-\delta$ associated with prior formation of γ' phase in Ni-Nb-V ternary alloys [16]. Kusabiraki et al. [17] also reported the similar results in Ni-Nb-Fe-Cr quaternary system. Therefore, it is likely that the metastable $\text{Ni}_3\text{Nb}-\gamma'$ phase tends to form with increasing Nb/Fe ratio in Fe-20Cr-40Ni-2Nb alloy.

In the present quaternary system, four intermetallic phases are found to exist, and the phases in equilibrium with γ phase change, depending on the alloy composition as well as temperature. Figure 8 shows the schematic illustrations of the phase equilibria in the quaternary system as functions of Ni and Nb content at 20% Cr. At 1473 K, all four intermetallic phases can be in equilibrium with the γ phase (Fig. 8(a)). At 1073 K, however, only δ and C14 phase becomes in equilibrium with γ phase (Fig. 8(b)). The change in the phase equilibria with temperature can be attributed to the occurrence of the following two consecutive transition peritectoid-type reactions:



At 1473 K, the γ phase is in equilibrium with μ phase, so that there would exist three three-phase tie triangles of $(\gamma+\delta+\text{C15})$, $(\gamma+\text{C15}+\mu)$ and $(\gamma+\mu+\text{C14})$. With decreasing the temperature,

the two three-phase tie triangles of ($\gamma+\delta+C15$) and ($\gamma+C15+\mu$) come closer and together, resulting in the four phase equilibria of ($\gamma+\delta+C15+\mu$). Then, it would split into the other combination of two three-phase tie triangles of ($\gamma+\delta+\mu$) and ($\delta+C15+\mu$), through the first peritectoid-type reaction (I). This is because, as far as the published data is concerned [9], the solid solution field of C15 Laves phase shown in Fig. 6 contracts more obviously with decreasing temperature, in comparison to that of the δ solid solution field. This means that C15 phase field in Fig. 8(a) moves left to more extent than the δ phase field does. And next, with further decreasing temperature, two three-phase tie triangles of ($\gamma+\delta+\mu$) and ($\gamma+\mu+C14$) come together and split into the other two three-phase tie triangles of ($\gamma+\delta+C14$) and ($\delta+\mu+C14$) in a similar manner through the second peritectoid-type reaction (II). These reactions would be responsible for the reason why the γ phase can become in equilibrium with only δ and C14 phases at lower temperatures.

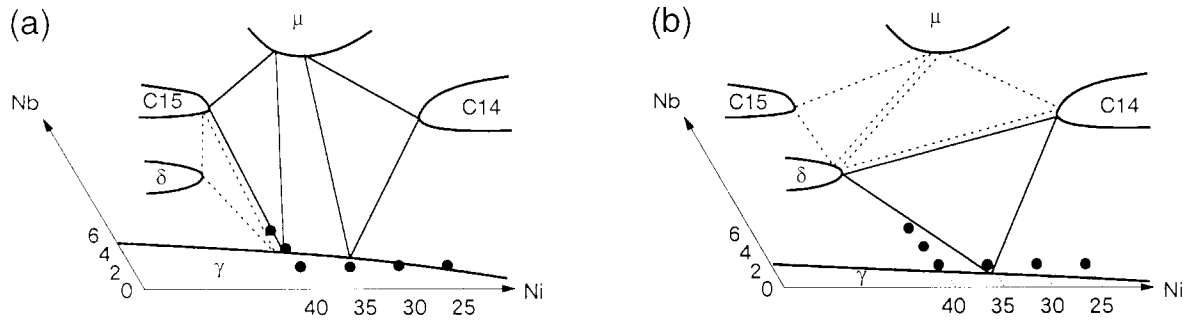


Fig. 8 Schematic illustration showing the phase equilibria as functions of Ni and Nb content at 20 % Cr in Ni-Nb-Fe-Cr quaternary system at (a) 1473K and (b) 1073K.

Summary

Phase equilibria in Ni-Nb-Fe ternary and Ni-Nb-Fe-20Cr quaternary systems at elevated temperatures was examined. In the ternary system at 1473 K, $Ni_3Nb-\delta$ ($D0_a$) single phase field expands along the equi-niobium concentration up to 10% Fe, and the δ phase becomes in equilibrium with $(Fe, Ni)_2Nb-\epsilon$ (C14 Laves) phase with a composition of Ni-30Fe-26Nb, and γ phase with its composition of Ni-35Fe-10Nb. Against expectations, there exists no $\delta+\gamma+\epsilon$ three-phase coexisting region. Instead, a new intermetallic phase with ordered hexagonal structure (hP24 with stacking sequence of abcbcb) was found to exist at compositions around Ni-22Nb-20Fe. In the quaternary system at 1473 K, four stable intermetallic phases of δ , ϵ (C14), C15 Laves (Cr_2Nb) and $(Ni, Fe)_6Nb_7-\mu$ phases exists, and the phase in equilibrium with γ phase changes in the following sequence of δ , C15, μ , C14 with decreasing the Ni/Fe ratio of the bulk alloy composition. However, at 1073 K, only δ and C14 phase become in equilibrium with γ phase. The change in the phase equilibria with temperature is attributed to the occurrence of two transition peritectoid-type reactions: (I) $\gamma + C15 \rightarrow \delta + \mu$, (II) $\gamma + \mu \rightarrow \delta + C14$. With increasing the Nb/Fe ratio, the γ/δ morphology changes from Widmannstätten type to lamellar type, indicating the formation of metastable $Ni_3Nb-\gamma''$ phase prior to that of stable δ phase due to the discontinuous precipitation.

Acknowledgements

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