

CRITICAL ASSESSMENT OF THE Fe-Ni-Ti SYSTEM

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Introduction

Fe-Ni-Ti is one of the ternary subsystems of the Al-Fe-Ni-Ti quaternary, the core system in the COST 535 (THALU) European Project.

As a starting point for a combined experimental and computational investigation of the system, the literature data concerning Fe-Ni-Ti and its binary subsystems have been collected and critically assessed.

Using the CALPHAD type approach, the Fe-Ni system was thermodynamically assessed by [1993Lee], the Fe-Ti system was critically assessed by [1991Mur] and thermodynamically modelled by several authors (the last one was [1999Kum]), the Ni-Ti system was also thermodynamically modelled by [1996Bel].

A review of the ternary system was previously published by [1990Gup] and recently updated by the same author [2001Gup], considering only literature up to 1994.

In the present work existing assessments are considered and updated using recent experimental information. Phase equilibria, crystal structure data and thermodynamic properties are discussed and selected material properties are briefly summarised.

Solid Phases

<i>Phase name, structure type, space group</i>	<i>Wyckoff posit.</i>	<i>Occup.</i>	<i>Coord. type</i>	<i>Sublattice model</i>	<i>Temperature (°C) and composition range(s) of stability</i>	<i>Lattice parameters (pm)</i>	<i>Comments</i>
A1 (fcc) cF4-Cu Fm-3m				(Fe,Ni,Ti) ₁ (Va) ₁ Empty sublattice accounts for interstitial sites.			Continuous solid solution in Fe-Ni extending into the ternary. Ti solubility increases with Ni content.
A2 cI2-W Im-3m				(Fe,Ni,Ti) ₁ (Va) ₃ Empty sublattice accounts for interstitial sites.			2 non-contiguous stability ranges: alpha-delta-Fe and beta-Ti.

A3 hP2-Mg P6 ₃ /mmc				$(\underline{\text{Fe}}, \underline{\text{Ni}}, \underline{\text{Ti}})_1(\text{Va})_{0.5}$ Empty sublattice accounts for interstitial sites.		Very limited Fe or Ni solubility in alpha-Ti.
B2 cP2-CsCl Pm-3m				$(\underline{\text{Fe}}, \underline{\text{Ni}}, \underline{\text{Ti}})_1(\underline{\text{Fe}}, \underline{\text{Ni}}, \underline{\text{Ti}})_1(\text{Va})_3$ Empty sublattice accounts for interstitial sites. Ordered form of A2.		Continuous solid solution between (FeTi) and (NiTi). The range of Ti solubility slightly increases with Ni content.

C14				<u>(Fe Ni Ti)</u> <u>(Fe Ni Ti)</u>			Binary TiFe solid solution

Crystal structure data and stability ranges are taken from [2000Oka] and [1995VPO] unless different reference is indicated.

Predominant elements in sublattice models are underlined.

Abbreviations in the Coordination column are: fcc = face centred cubic, hcp = hexagonal close packed, bcc = body centred cubic, ico=icosahedral, F-K = Frank-Kasper, oct = octahedral, cmp = complex

Isothermal sections

Van Loo et al. [1981Loo] provided an isothermal section of the whole system at 900°C which is redrawn in **Fig. 1**. The invariant points in the limiting binaries were plotted, according to the present assessments of the experimental diagrams, with solid circles. This figure shows that there is no ternary compound while the binary compounds present significant substitution of Fe for Ni and vice versa. In this figure, empty circles correspond to the composition of the phases determined experimentally (read off the provided figure and listed in **Table 1**), they are linked with dashed tie-lines. Three phase triangles shown with dashed lines as well as single phase fields were guessed by Van Loo et al. [1981Loo] from their data. The A1+Fe₂Ti+Ni₃Ti three phase region is shifted toward higher Fe content with respect to the tentative isothermal section proposed by Vogel and Wallbaum [1938Vog] from their isopleth sections (see below). As pointed out previously by Gupta [1991Gup], this shift is in agreement with data from Speich [1963Spe] and Fournelle [1979Fou] who showed that an alloy with 28.5 at. % Ni and 6.8 at. % Ti is single phase A1 (γ -FeNi) at 1100°C but two-phase A1+Ni₃Ti at 900°C. This alloy appears as a greyed triangle in **Fig. 1**.

Table 1: tie-lines taken off the 900°C isothermal section [1981Loo].

phases in equilibrium	Phase 1 Ni (at. %)	Phase 1 Ti (at. %)	Phase 2 Ni (at. %)	Phase 2 Ti (at. %)
A1(γ -FeNi)+Ni ₃ Ti	82.2	5.5	73.6	24.5
	60.5	5.5	70.7	24.1
	25.6	6.5	64.6	23.6
Ni ₃ Ti+B2(FeTi)	73.6	25.5	50.7	46.2
B2(FeTi)+NiTi ₂	44.1	51.7	32.0	66.0
	34.6	51.4	25.4	65.8
	21.2	51.9	19.1	65.8
	6.6	52.4	13.7	66.0
NiTi ₂ +A2(β -Ti)	22.7	67.0	6.5	87.7
B2(FeTi)+Laves phase	24.0	47.2	12.5	34.2
	13.7	48.6	5.6	34.0
Laves phase+A2 (α_{Fe})	3.4	27.8	1.4	4.7
Laves phase+A1 (γ -FeNi)	5.6	27.4	5.7	4.2
Laves+Ni ₃ Ti	19.8	30.2	62.3	25.0
	17.0	28.3	62.3	24.5

Abramychева et al. [1999Abr] realized diffusion couples at 1000°C from which they drew an isothermal section reproduced in **Fig. 2**. As before, solid dots are invariant points taken from the assessed binary diagrams. Compositions of the phases in equilibrium were read from the provided tables and represented as empty circles and squares for two phase and three phase equilibria respectively. The main features of this diagram are similar to the one drawn by Van Loo et al. [1981Loo], though a number of differences are stressed below.

At Ti content lower than 50 at%, a number of data have been reported on **Fig. 2** in addition to the results from Abramycheva et al. [1999Abr]. As before, the greyed triangle corresponds to the alloy studied by Speich [1963Spe] and Fournelle [1979Fou]; Speich found the solvus at 982°C [1963Spe]. Close to this triangle, the greyed square represents the solubility limit of Ti in A1- γ FeNi determined by Abraham [1969Abr] at 1027°C on an alloy containing 27 at.% Ni. It is seen that these two points are at Ti contents significantly lower than the boundary limit reported by Abramycheva et al. [1999Abr]. This scattering of the solubility limit of Ti in A1 has been previously discussed by Gupta [1991Gup] who mentioned also the work by Drake [1982Dra] in which the maximum solubility of Ti was as high as 12 to 13 at. %. The boundary of the A1 phase field has been redrawn with respect to Abramycheva et al. [1999Abr] to look more similar that by Van Loo et al. [1981Loo] and the three phase (Laves phase+Ni₃Ti+A1) triangle has been adjusted accordingly.

At the right of the lower part of **Fig. 2**, a tie-line between A1(γ -FeNi) and Ni₃Ti from Jia et al. [1994Jia] is shown with empty triangles. The Ti content of this latter phase appears quite low with respect to the accepted values in the binary system which are due to the same author. Also, in the middle of the composition triangle, the three phase (Laves phase+B2+Ni₃Ti) triangle has been slightly adjusted to agree with the experimental set of tie-lines. One should notice also that the ternary extensions of the phase fields of Ni₃Ti and Laves phases turn to lower and higher Ti contents in the 900°C and 1000°C isothermal sections respectively.

In the Ti-rich part, the main difference with the section at 900°C is the presence of a liquid field that is expected from the knowledge of the binary Ni-Ti system. In agreement with Van Loo et al. [1981Loo], Abramycheva et al. [1999Abr] observed a three phase A2(β -Ti)+B2+NiTi₂ equilibrium on the Fe-Ti side. In order to agree with the isopleths established by Alisova et al. [1994Ali] and described in the next section, Abramycheva et al. [1999Abr] suggested that the stability of NiTi₂ at 1000°C should be limited to an area close to the Fe-Ti side while only β_{Ti} and B2 would be in equilibrium at lower Fe content along the line at 33 at% Ti. This seems quite unlikely and other results showing that NiTi₂ may be stable at higher temperature and intermediate Fe contents lead to disregard the work by Alisova et al. [1994Ali]. This conclusion has been used to modify the Ti rich corner of the 1000°C isothermal section with respect to the original work by Abramycheva et al. [1999Abr].

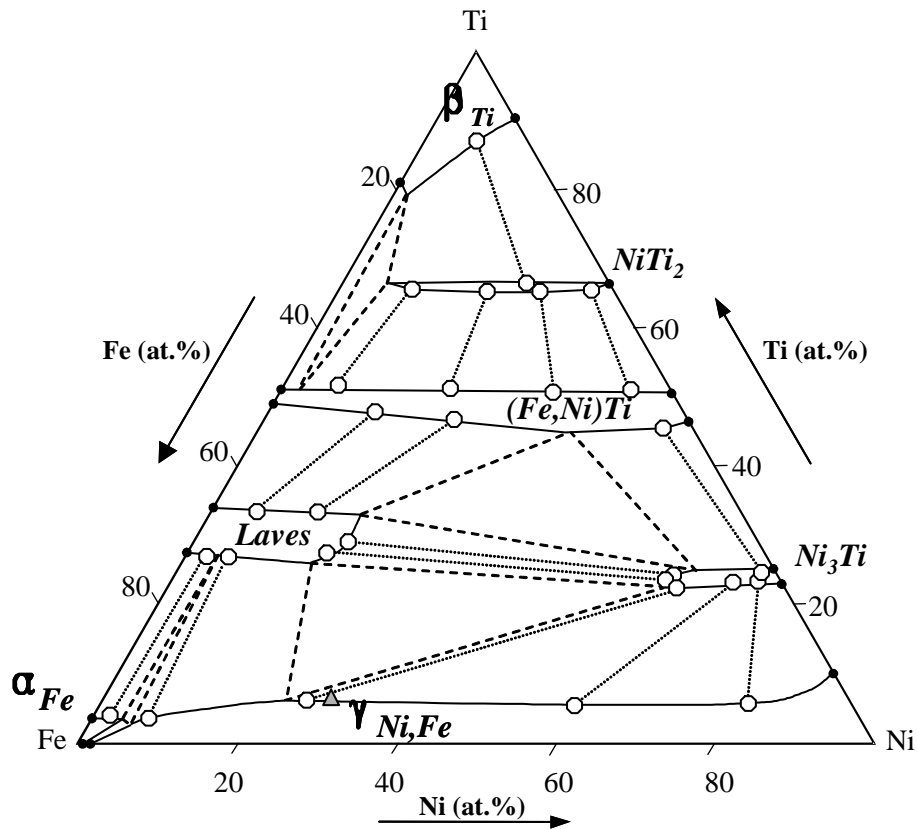
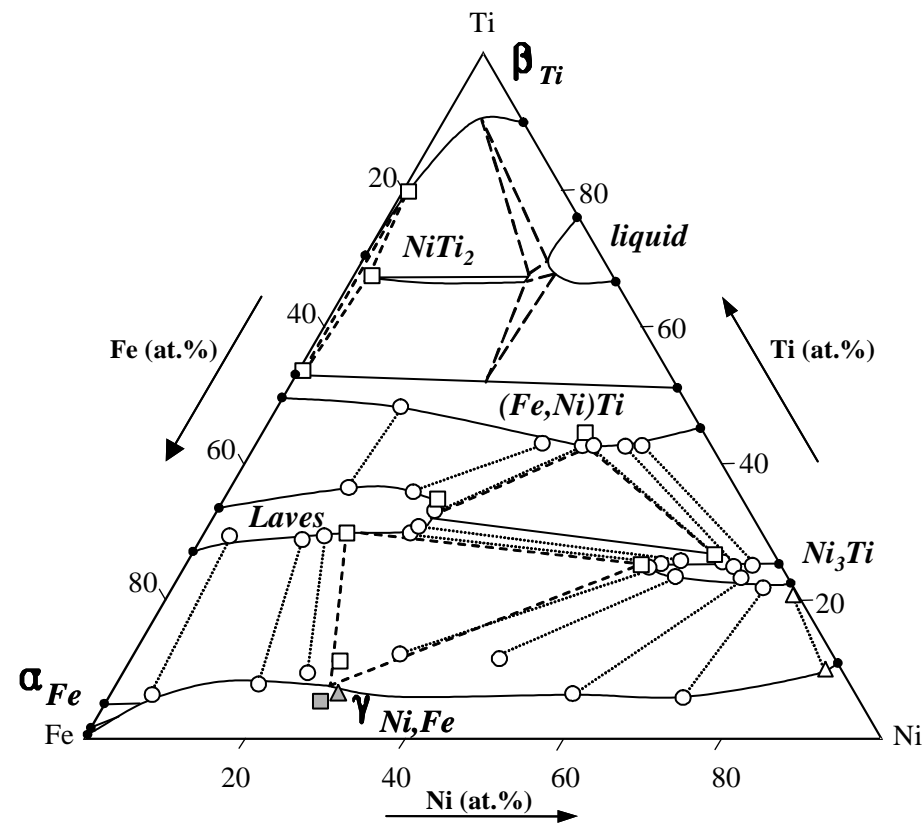


Figure 1: isothermal section at 900°C according to Van Loo et al. [1981Loo]. Solid circles represent phase compositions according to the accepted assessments of the binary systems. Empty circles are experimental data [1981Loo] and greyed triangle represents the composition of a two-phase A1(γ)+Ni₃Ti alloy according to [1979Fou].

Figure 2: isothermal section at 1000°C, mainly according to Abramycheva et al. [1999Abr]. Solid circles represent phase compositions according to the accepted assessments of the binary systems. Empty circles are experimental data. A few other results have been added which are described in the text.



Liquidus projection

Vogel and Wallbaum [1938Vog] studied the Fe-Ni-Ni₃Ti-Fe₂Ti part of the system. They performed thermal analysis and microstructural investigation on several tens of alloys and reported their results as isopleth sections. From these, they could draw a partial liquidus projection that shows the Ni₃Ti-Fe₂Ti line to be quasi-binary. Three invariant points, e, E and U₁, were thus established that are listed in **Table 2**. **Fig. 3** presents the liquidus projection, in which binary invariant points (solid circles) are from the presently accepted assessment of the corresponding systems. Four points along monovariant lines could be read off the isopleth sections of Vogel and Wallbaum, they are listed in **Table 3** and plotted with empty circles in **Fig. 3**. In his review, Gupta [1991Gup] emphasized that the binary peritectic point in the Fe-Ni system used by Vogel and Wallbaum was located at too high Ni content. Accordingly, Gupta moved the ternary peritectic point U₁ closer to the Fe-Ni side of the composition triangle. In the present diagram, this point was shifted to even lower Ni content because the isopleth section at Fe:Ni equal to 90:10 in the original work of Vogel and Wallbaum [1938Vog] does not show any thermal arrest that could be related to crossing of the line p₁U₁.

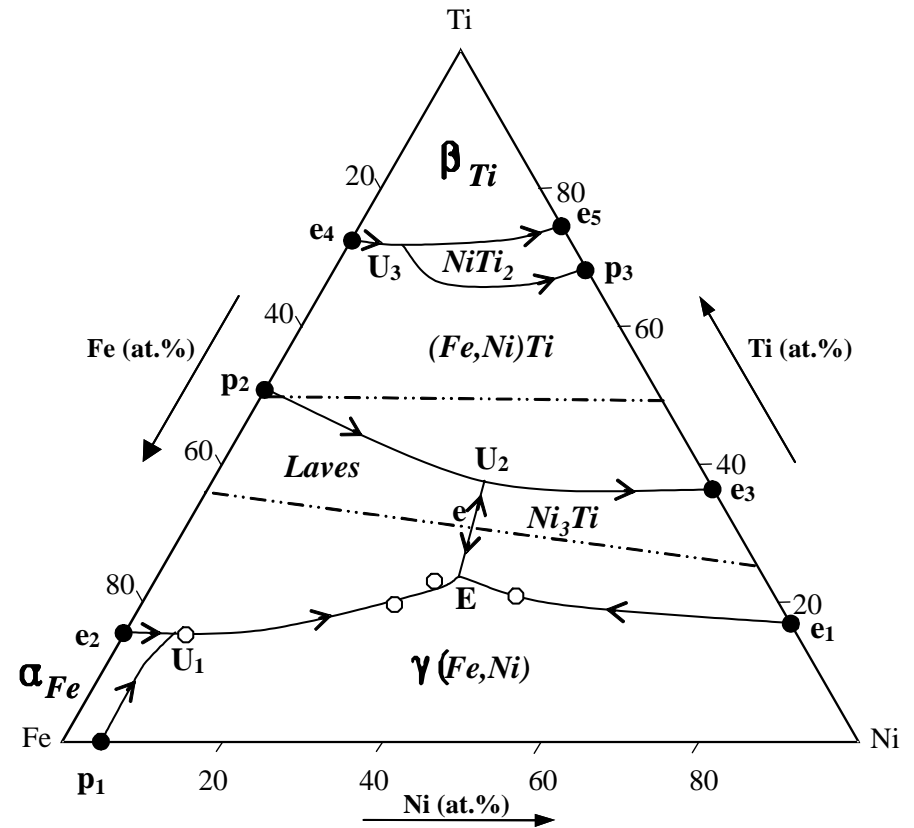
Table 2: Ternary invariant points on the liquidus surface.

Reaction	Temperature (°C)	phases
e	1320	Liquid+Ni ₃ Ti+Laves phase (Fe ₂ Ti)
U ₁	1200	Liquid+A1 (γ-FeNi)+A2 (α _{Fe})+Laves phase (Fe ₂ Ti)
E	1113	Liquid+A1(γ-FeNi)+Ni ₃ Ti+Laves phase (Fe ₂ Ti)
U ₂		Liquid+Ni ₃ Ti+Laves phase (Fe ₂ Ti)+B2(FeTi)
U ₃	960°C	Liquid+A2(β _{Ti})+B2(FeTi)+NiTi ₂

Table 3: points along monovariant lines according to [1938Vog].

isopleth section	Ni (at. %)	Ti (at. %)
Fe:Ni = 90:10	8.1	15.3
Fe:Ni = 40:60	46.5	21.1
Fe:Ti = 74:26	31.9	19.8
Fe:Ti = 65:35	35.2	23.2

Figure 3: Liquidus projection of the Fe-Ni-Ti. Solid lines are monovariant lines, interrupted lines show quasi-binary sections.



Complete solubility of Fe and Ni in the ordered B2 phase has been evidenced by Dudkina and Kornilov [1967Dud]. This defines a second quasi-binary section of the diagram although the B2 phase results from a peritectic reaction on the Fe-Ti side. Dudkina and Kornilov [1967Dud] determined the solidus along this line but neither the liquidus nor the three phase equilibria at the Fe-Ti side. The solidus shows a minimum at about equal amount of Fe and Ni which should correspond to a congruent melting point as stressed by Gupta [1991Gup]. The monovariant line p_2-e_3 and point U_2 in the middle of the composition triangle, as well as the monovariant lines and point U_3 in the Ti-rich corner, were tentatively located by Gupta [1991Gup].

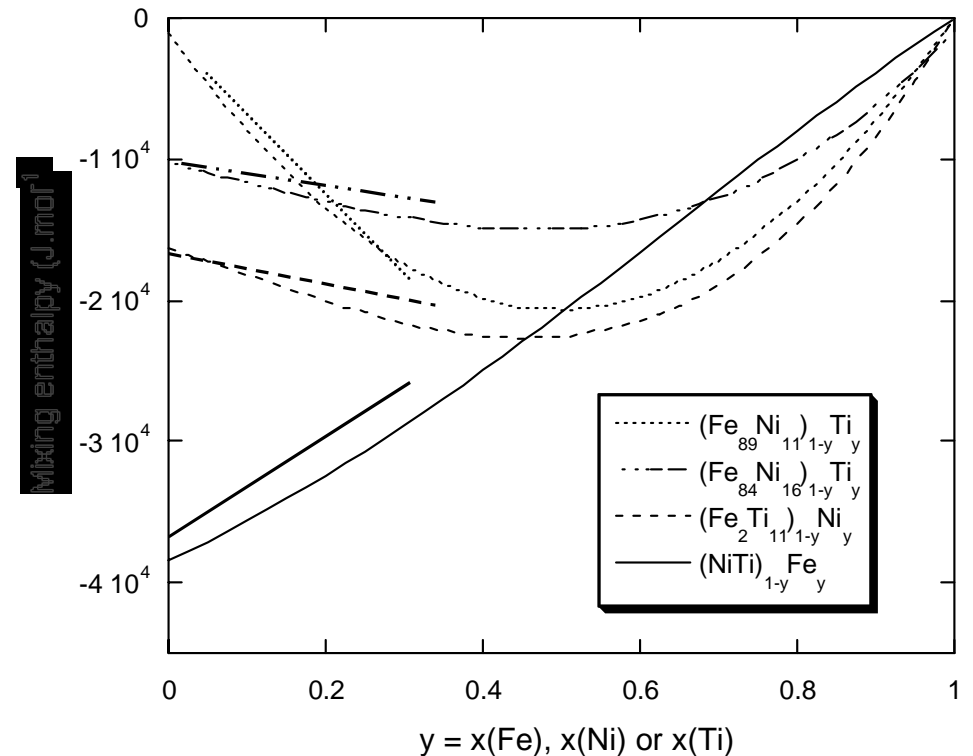
More recently, Gupta [2001Gup] reassessed his former liquidus projection to account for the work of Alisova et al. [1994Ali] who provided three vertical sections of the diagram in the Ti rich region. These sections were selected with Fe:Ni equal to 3:1, 1:1 and 1:3 respectively. Alisova et al. [1994Ali] found that only $A2(\beta_{Ti})$ and B2 appear in the first two sections at temperature higher than 700°C, while $NiTi_2$ may also be in equilibrium with the liquid phase in the last section. Following Alisova et al. [1994Ali], Gupta [2001Gup] moved point U_3 in **Fig. 3** to much lower Fe content than in his first assessment. However, this author did not notice that these results are in disagreement with the isothermal section at 900°C provided by Van Loo et al. [1981Loo] who showed $NiTi_2$ to nearly cross the composition triangle by substitution of Ni by Fe (**Fig. 1**).

As pointed out above, Abramychева et al. [1999Abr] found also that $NiTi_2$ can be in equilibrium with $A2(\beta_{Ti})$ and B2 very close to the Fe-Ti side of the composition triangle and attempted to reconcile this result with the isopleth sections proposed by Alisova et al. [1994Ali] by limiting the stability of $NiTi_2$ at 1000°C to a small domain close to the Fe-Ti side. However, Efimenko et al. [2000Efi] reported results that disagree also with the isopleth sections published by Alisova et al. [1994Ali]. Efimenko et al. [2000Efi] made diffusion couples by melting a mixture of Ni and Ti powders (in the ratio corresponding to eutectic e_3) over an iron substrate maintained at either 1100, 1200 or 1250°C for times in the range 5 to 120 min. The samples were then cooled and analysed by EPMA. During the treatments at high temperature, the iron content of the melt reached rapidly a constant value varying from 11.6 to 20 at. % at increasing temperature while the Ni:Ti ratio remained the same. The two phases $NiTi_2$ and B2 were observed in every case while $A2(\beta_{Ti})$ would have been observed according to Alisova et al. [1994Ali]. It thus seems wise to disregard the results by these latter authors and to consider that $NiTi_2$ is stabilized by Fe to such an extent that it could form peritectically (or congruently?) from the liquid phase far inside the composition triangle. This conclusion has been used in drawing the monovariant lines in the Ti corner in **Fig. 3**.

Thermodynamic data

Thiedemann et al. [1999Thi] have measured enthalpy of mixing in the Ni-Ti and Fe-Ni-Ti liquid by levitation calorimetry. Their results for the Ni-Ti binary system are in close agreement with previous data in the Ni-rich part, they are the only ones available in the Ti-rich part. The authors could represent conveniently their results following the regular associate model considering that associates of composition Ni_3Ti are formed. In the ternary system, the authors performed experiments along part of three lines in the composition triangle. Assuming no ternary interactions and using previous assessment of the Fe-Ni and Fe-Ti systems, they could reproduce the main features of their results as well as the experimental data from Lück et al. [1991Lüc]. On the basis that ternary interactions are not significant, calculations of the mixing enthalpy along the lines experimentally explored by Lück et al. [1991Lüc] and Thiedemann et al. [1999Thi] were performed with the CALPHAD-type data available. These calculations are shown in **Fig. 4** where they are compared with the experimental data. It is observed that the main features of these results are reproduced.

Figure 4: comparison of calculated enthalpy of mixing with experimental results from Lück et al. [1991Lüc] and Thiedelmann et al. [1999Thi] shown as straight segments.



Application Properties

The by far most important technical applications for Fe and Ni are of course steels and superalloys [1998Bür]. Additions of Ti are important in steels to form carbides, as grain refining element or to enhance oxidation resistance. However, only small quantities of up to 2wt.% are needed. Of greater importance are additions up to 5 wt% Ti to Ni-base superalloys where Ti forms the γ' phase together with Ni and Al, which is decisive for the high creep strength of this class of alloys. However, the properties of the alloy classes are controlled by the combined influences of all elements, which sometimes exceeds the number of 10. Applications being more focussed on the Fe-Ni-Ti system are complex metallic alloys such as shape memory alloys, bulk metallic glasses, Heusler phases, quasicrystals or hydrogen storage intermetallic compounds.

Shape memory alloys are metallic materials showing a stress induced diffusionless phase transformation from the high temperature phase austenite into the low temperature phase martensite [1986Hor]. The strain related to this phase transformation is fully reversible after removing the stress. Typical shape memory alloys are based on NiTi alloys. The most known technical alloy is called “Nitinol” having a composition of 55-55.5wt% Ni – 45-45.5wt% Ti with small additions of Co to vary the M_s temperature [1992Por]. This alloy shows a fully reversible transformation of one ordered phase into another at 170°C resulting in a shear transformation of up to 16%. The M_s temperature is very sensitive to alloy composition which has to be close to 1:1 ratio of Ni and Ti, however, tolerating small overstoichiometry of Ni. Too high Ni contents will result in a precipitation of Ni_3Ti . An interesting feature of this transformation is the abrupt change of Young’s modulus and yield strength, being utilised in technical applications. Such applications are in medicine, for instance surgery instruments or endoscopes [www.memory-metalle.de], but also in construction industry as fasteners [2001Hsu; 2001Kri]. A most recent overview about status of development [2004Fir], properties [2004Egg], applications [2004Mor] and characterisation methods [2004Sch] of NiTi and other shape memory alloys is given in the complete issue 1-2 of Materials Science and Engineering A, vol. A378 (2004).

Bulk metallic glasses (BMG) are formed in various alloy system among which Fe/Ni-(Zr-Hf-Nb)-B, Ni-Zr-Ti-Sn-Si, Ti-Ni-Cu-Sn and Ti-Zr-Fe are of interest in this connection. Important factors controlling the glass forming ability are a (i) multicomponent system consisting of more than three elements, (ii) a significant atomic size mismatch above 12%, and (iii) negative heats of mixing [2002Ino]. Technical applications of BMG are driven by their extreme mechanical and physical properties, for instance high strength exceeding 2GPa, low damping, high corrosion resistance or special magnetic properties [2002Ino; 2003Nis].

Heusler phases are intermetallic phases based on the prototype Cu_2MnAl and show ferromagnetic behaviour though not containing Fe or Ni. They also form in other alloying systems such Ni_2AlX ($X=Ti, Cr, V, Mn$) or X_2AlTi ($X=Fe, Co, Ni, Cu$) [2002Ish] and show some interesting physical properties due to their electronic structure such as semiconductivity [2003Pal], thermoelectricity [2001Gol] and magnetic properties [2003Nan]. This might be interesting for new technical applications in these fields.

Quasicrystals were discovered in 1984 in the Al-Mn system and are characterised by a non-periodic lattice, however showing long-range order. They form in several alloying systems, of special interest in this case are Ti-Zr-Ni [2004Qia] and Ti-Ni-Fe-Si [1989Bah] Their atomic structure results in interesting mechanical properties such as high hardness and elasticity coupled with reasonable ductility, as well as in exceptional electronic properties. These properties are already applied in quasicrystalline coatings with high abrasion resistance and might be used in thermoelectricity in future.

Metallic materials for hydrogen storage are either hydride forming transition metals MH_n ($n=1, 2, 3$), metallic hydrides of intermetallic compounds AB_xH_n ($x=0.5, 1, 2, 5$) or complex hydrides forming an ionic or covalent compound upon hydrogen absorption [2003Zue]. The intermetallic compounds to form hydrides consist of a hydride forming element A (rare earth or alkaline earth) and a transition metal B forming unstable hydrides. Among the B metals Ni is often included, since it is an excellent catalyst for hydrogen dissociation. These compounds can absorb large amounts of hydrogen at a constant pressure and their properties can be tailored by partial substitution of the constituent elements in the host lattice [1988Sch]. In case of the Fe-Ni-Ti system compounds with high hydrogen absorption are TiFe with partial substitution of Fe with Ni [1983Ogu] and Ti_2Ni [2003Zue]. Technical applications of hydrogen storage materials have been found in Ni metal hydride (Ni-MH) batteries since the 1990s. Upcoming applications are hydrogen tanks for fuel cell cars because of their compactness and safety for hydrogen energy storage. However, the storage capacity and weight has to be improved. A lot of research work about AB_xH_n intermetallic compounds has been made in the past [1984Gri; 1988Sch]. However, most promising candidates for future hydrogen storage materials are complex hydrides formed by light elements. Because of their low density and high storage capacity they are ideal candidates for mobile applications and there is a whole field of new compounds to be explored [2003Zue].

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