The Fe-Ni (Iron-Nickel) System

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Equilibrium Diagram

The equilibrium phases of the Fe-Ni system are: (1) the liquid, L ; (2) the bcc, high-temperature (δ Fe) solid solution; (3) the fcc (yFe,Ni) solid solution; (4) the bcc, low-temperature (αFe) solid solution; and (5) the FeNi₃ intermetallic compound, which forms by a first-order order-disorder transformation below 517 *C and has an extended range of homogeneity.

Early systematic efforts to construct a phase diagram for Fe-Ni were made by [1899Osm], [05Gue], [10Heg], and [10Rue]. Evaluated phase diagrams for the Fe-Ni system were given previously by [38Mar], [Hansen], [Shunk], and [82Kub]. The present assessed Fe-Ni equilibrium diagram is shown in Fig. 1.

Llquldus and Solidus

The liquidus and solidus for Fe-rich alloys up to about 12 at.% Ni were measured by [57Hel] using thermal analysis. [23Han] measured the liquidus and solidus over the entire range of composition. Measurements were also made by [05Gue], [25Kas], [31Ben], [37Jen], [25Vog], [27Vog] and [28Vog]. The data are summarized in Table 1. According to [23Han], the minimum in the liquidus curve is located at 1438 *C (temperatures quoted throughout were converted to IPTS-68) and about 67 at.% Ni. According to [37Jen], the minimum is between 1422 and 1427 *C. Based on a least-squares fit taking into account both the measured boundaries and available thermodynamic data (see Thermodynamics), we find the most probable location for the minimum to be 1440 *C and 66.0 at.% Ni, in good agreement with [23Han].

[64Hum] calculated the Fe-rich liquidus/solidus based on an ideal-solution model. This procedure gives too large a separation between the liquidus and solidus when extended to Ni-rich alloys. The liquidus and solidus separations of Fig. 1 are based primarily on those predicted by the measured thermodynamic parameters in the liquid and fee phases, with the assessed boundary location giving greatest weight to the measurements of the liquidus. These boundaries are close to the predictions of [85Tom], which were based on thermodynamic parameters measured using mass spectrometry. They are shown on an expanded scale in Fig. 2.

Phase Boundaries

Measurements of the (α Fe/ δ Fe) phase boundaries were made by [20Han], [23Han], [25Kas], and [57Hel]. Comparisons with thermodynamic measurements (see below) and with liquidus and solidus measurements indicate a narrow two-phase $(\alpha Fe) + (\delta Fe)$ region and a narrow peritectic reaction--L + (δ Fe) \leftrightarrow (γ Fe,Ni) at 1514 \pm 2 °C and 3.5, 4.9, and 4.2 \pm 0.5 at.% Ni, respectively. Measured values are listed in Table 1 and compared with the equilibrium diagram in Fig. 3.

(αFe)/(γFe, Ni) Phase Boundaries

Due to the sluggishness of the (γ Fe,Ni) \rightarrow (α Fe) and (α Fe) \rightarrow (YFe , Ni) phase transformations below 800 °C, the (αFe)/(YFe , Ni) equilibrium boundaries are difficult to determine (see Metastable Phases). [36Jet], [39Owe], [41Owe], and [49Owe] used powder X-ray diffraction (XRD) techniques down to 300 "C. [65Gol] used electron microprobe techniques to measure concentration profiles on samples equilibrated at temperatures as low as 500 "C. [80Rom] used a scanning transmission electron microscope (STEM) on samples equilibrated between 670 and 300 $^{\circ}$ C. These results, along with those of [69Sta], demonstrated clearly the retrograde solubility of Ni in (α Fe). The data of [49Owe], [49Jon], [65Gol], and [80Rom] agree fairly well at 500 *C and above. The boundaries shown in Fig. 1 were constructed using these data, giving greatest weight to the results of [80Rom]. Numerical values determined by the various investigators are given in Table 2. Figure 3 shows the assessed boundaries and compares them with the experimental data. Enthalpies of the $(\alpha Fe) \rightarrow (\gamma Fe, Ni)$ transformation were measured by [26Kaw], [36Sam], [37Koe], [40Zui], [59Sch], and [67Hil].

FeNI3

Evidence for ordered FeNi₃ was obtained first by [32Dah], [33Dah], [36Dab], [39Kus], and [39Lee] using XRD. Because of the nearly equal X-ray scattering factors for Fe and Ni, the ordering is difficult to observe with X-rays (see [38Haw]). The results of [39Lee] were confirmed by [39Haw], also with X-rays. Variations in the magnetic properties, electrical resistance, and hardness with heat treatment of alloys showing FeNi3 order were investigated by [32Dab], [33Dab], and [36Dah]. Based on X-ray and electron microscope observation of annealed thin films and on the results of $[52Hun]$, $[63Heu]$ concluded that the $(\gamma Fe,Ni)$ phase decomposes eutectoidally to (α Fe) and FeNi₃ at 345 °C and 52 at.% Ni, a conclusion that still appears valid. (The existence of a eutectoid near this same point based on the magnetic transition **had** been postulated earlier; see [21Mer].)

The critical behavior of the temperature derivative of the resistivity around the FeNi₃ composition was investigated by [82Ore]. No anomaly in the resistivity was found at the order-disorder temperature, but one was found at the Curie temperature. The phase diagram in the composition range 69 to 77 at.% Ni and in the temperature range around 510 "C was studied in considerable detail by [80Van] and [81Van] using Mössbauer spectroscopy. They explained a 15 °C hysteresis zone between ordus and disordus as a magnetic effect. [81Lef] investigated the local atomic arrangement in FeNi₃ using neutron diffuse scattering and a single crystal quenched from four different temperatures. [72Cal] found that the order-disorder transformation of FeNi3 is not second order and observed a two-phase zone of 5 or 6 °C for alloys between 71 and 75 at.% Ni. On the basis of a calorimetric study, [52Iid] and [54Iid] concluded that short-range order forms in FeNi3 before long-range order is observable by other methods.

The ordering behavior of FeNi₃ was also investigated by [37Kal], [39Haw], [39Kay], [40Nix], [48Kal], [50Jos], [53Rhi], [53Gei], [57Lya], [57Vit], [58Kus], [62Kac], [63Dav], [63Tre], [65Gon], [72Cal], [73Cal], [75Bil], [75Leb], [77Hut], [79Des], and

Table 1 High-Temperature Phase Boundary Data for Fe-Ni Alloys

Note: All results corrected to IPTS-68 and to agree with 1538 °C as the melting temperature of pure Fe and 1455 °C as the melting temperature of pure Ni.

| Composition, | Temperature, 'C | | | | | |
|--------------|-----------------|----------------|--|---|--|--|
| at.% Ni | Liquidus | Solidus | $(\delta Fe)/(\delta Fe) + (\gamma Fe N i)$ | $(\delta Fe) + (\gamma Fe,Ni)/(\gamma Fe,Ni)$ | | |
| From [37Jen] | | | | | | |
| | \cdots | 1479 | | \cdots | | |
| | \cdots | 1458 | | | | |
| | \cdots | 1447 | | \cdots | | |
| | \ddotsc | 1441 | \cdots | \cdots | | |
| | \cdots | 1440 | \cdots | \cdots | | |
| | \cdots | 1439 | \cdots | \cdots | | |
| | \cdots | 1418 | | \cdots | | |
| | \cdots | 1423 | | \cdots | | |
| | \cdots | 1438 | | \cdots | | |
| From [57Hel] | | | | | | |
| | 1528 | 1526 | 1455 | 1462 | | |
| | 1524 | 1520 | 1484 | 1483 | | |
| | 1520 | 1514 | 1503 | 1506.5 | | |
| | 1516 | 1511.5 | 1514 | \cdots | | |
| | 1513.5 | 1510 | | \cdots | | |
| | 1508.5 | 1503.5 | | \cdots | | |
| | 1502.5 | 1598.5 | | | | |
| | 1497 | 1491 | \cdots | \cdots | | |

Table 1 High-Temperature Phase Boundary Data for Fe-Ni Alloys (continued)

[84Lef]. Experimental values reported by various investigators are listed in Table 3 and compared in Fig. 4.

Metastable Phases

At low temperature (under about 800 °C), the $(\alpha Fe) + (\gamma Fe, Ni)$ field is relatively broad, and attainment of equilibrium involves considerable diffusion. Diffusion rates at these lower temperatures are low; consequently, very long times are required to establish equilibrium, and normal conditions favor a diffusionless (or martensitic) transformation. This transformation exhibits considerable hysteresis. In early work, [20Han] used metallography to determine two sets of boundaries for the $(\alpha Fe) + (\gamma Fe,Ni)$ twophase region---one on heating, and one on cooling. The $(\gamma Fe, Ni)/(\alpha Fe) + (\gamma Fe, Ni)$ boundary determined in this way agrees closely with that determined by thermal analysis (the $(\alpha Fe)/(\alpha Fe) + (\gamma Fe, Ni)$ boundary is not detected by thermal analysis). [20Han] also concluded that the $(\alpha Fe) \rightarrow (\gamma Fe, Ni)$ transformation is accelerated by the presence of impurities.

From a practical standpoint, a diagram giving the details of this irreversible transformation is often of more importance than one giving the equilibrium boundaries in this region. Figure 5 exhibits experimental transformation measurements of [25Pes], [27Hon], [49Jon], and [56Kau] and compares them with the equilibrium boundaries. In this figure, the supersaturated bcc phase that results from the diffusionless transformation is denoted α_2 . The solid lines are estimates of A_s , M_s , A_f , and M_f , where A_s and M_s are the so-called austenite and martensite start temperatures (10 vol.% of the alloy having transformed), and A_f and M_f are the so-called austenite and martensite finish temperatures (90 vol.% of the alloy having transformed). These lines are valid only over a range of cooling and heating temperatures between approximately 2 and 150 °C/min. The uncertainties in the estimated A_s , A_f , M_s , and

Mf temperatures are large, but decrease as the Ni concentration decreases.

In the α_2 or (γ Fe,Ni) field, the alloy will be all (γ Fe,Ni) if it is being cooled from the (γ Fe,Ni) field or all α_2 if it is being heated from the α_2 field. Because the transformation from (γ Fe,Ni) to α_2 is diffusionless, it can occur at very low temperatures for alloys with greater than 30 at.% Ni. [56Kau] reported the M_s temperature of $(-223 °C)$ (50 K) for a 33 at.% Ni alloy. In addition, the M_s temperatures of various steels are known [54Mey, 48Fis, 74Ume, 63Yeo, 82Brol] to change with the temperature at which they are austenized. That this change is due to the effect of austenizing temperature on grain size was shown by [51Mac] and [74Ume]. For an Fe-31 at.% Ni alloy, [74Ume] found that the M_s temperature varied between 200 and 230 °C as the grain size varied between 5 and 70 μ m. Impurities and internal stresses also have an important effect on the transformation temperatures $[e.g. 82Rod]$.

The diffusionless character of the $\alpha_2 \rightarrow (\gamma Fe, Ni)$ transformation was shown clearly by [32Sch]. [34Deh] reported that the transformed alloy is bcc, and is not tetragonal as is Fe-C martensite, with the microstructural similarity of the two martensites being due to the similar way in which they transform from the γ phase. [35Sch] found that for greater than 10 at.% Ni, the (γ Fe,Ni) \rightarrow $(\alpha$ Fe) transformation temperature could not be lowered by long annealing times (up to 100 h). Some early diagrams, such as that of [36Mer], were drawn showing the irreversibility of the diffusionless transformation directly.

[62Gil] reported that the diffusionless transformation is massive type for Ni contents less than about 15 at.%. For very rapid cooling and/or for higher Ni concentration, [66Hum] and [62Gil] reported that the transformation is martensitic type.

According to [49Jon], the diffusionless $\alpha_2 \rightarrow (\gamma Fe, Ni)$ transformations are independent of the cooling or heating rate for rates between 2 and 150 $^{\circ}$ C/min. [76Ino] and [82Ray] showed that the

(a) Diffusion couple, analyze with electron probe. (b) Quench as α_2 anneal, analyze with electron probe. (c) Carbonyl vapor pressure.

martensitic transformation in Fe-24 wt.% Ni can be suppressed to below room temperature by splat cooling. For alloys with less than about 30 at.% Ni, the transformation is isothermal, and the transformation temperature is strongly dependent on impurity contents. Above 30 at.% Ni, the transformation is athermal and **less** dependent on impurities [83Kam]. Further investigations of the nature of the $\alpha_2 \rightarrow (\gamma Fe, Ni)$ transformation were made for example by [29Gos], [30Rob], [48Fis], [47Oel], [51Smo], [62Bre], [62Yeo], [63Gol], [67Rob], [71G-eo], [77Roi], [79Mat], [81Bor], [82Duf], [84Bor], [84Izm], and [84Rin].

Considerable supercooling of liquid Fe-Ni alloys is possible. [78Con] observed supercooling of up to 150 K in an alumina crucible for alloys between 6 and 90 at.% Ni. [83Abb] studied supercooling in levitation melted 25 at.% Ni samples, obtaining supercooling up to 270 *C, somewhat less than the 300 *C reported by [66Kat] for alloys surrounded by glass coatings. Transformation of (γ Fe,Ni) to (α Fe) in thin films was investigated by [78Gal].

Alloys containing approximately 20 to 50 at.% Ni-the so-called Invar alloys--exhibit anomalous thermomechanical and thermochemical behavior, including a region of very low coefficient

of thermal expansion. [79Cha] showed that these Invar anomalies tend to disappear in alloys that have been electron irradiated to enhance diffusion and thereby accelerate the approach to true equilibrium. Thus it is probable that the Invar anomalies are a characteristic of metastable alloys. Literature on the Invar state is extensive, and we refer here to [60Kon], [64Ban], [72Wei], [78Kat], [78Cha], and [79Shi], where many additional references may be found. Recently, [85Chu2] proposed that the presence and disappearance of the Invar anomalies are related to a spinodal gap due to a tricritical point arising from the magnetic transition in fcc alloys (see Fig. 6).

An orthorhombic phase in thin films with a composition in the $(\alpha \text{Fe}) + (\gamma \text{Fe}, \text{Ni})$ region of the equilibrium diagram was indicated by [58Pin]. This phase was not retained above 650 *C. [56Cec] postulated that small particles of 30 at.% Ni that were rapidly cooled from the liquid state could pass directly to the (αFe) phase. A surface phase, either orthorhombic or tetragonal, which formed on 50 at.% Ni magnetic tapes after 18 months at room temperature was reported by [59Ana]. A reversible transformation from one fee structure to two fee structures below about 150 *C in 30 to 45 at.% Ni alloys was reported by [61Ana]. Rapidly solidified Fe-Ni alloys were also studied by [73Miz], [79Bos], [84Gor], and [S4Miu].

Metastable FeNI and Fe3NI

The existence of ordered structures based on Fe₃Ni and FeNi have been proposed by a number of investigators. For the equiatomic composition, an order-disorder reaction below 321 ± 2 °C was reported by [60Paul, [62Paul], and [62Pau2] for alloys that were heavily irradiated by neutrons. [56Tin] gave X-ray data supporting the existence of an ordered structure near the Fe₃Ni composition. The existence of Fe3Ni and FeNi ordered structures was investigated further by [43Hos], [58Kus], [60Paul, [61Ban], [63Heu], [63Mar], [64Nee], [70Gro], [71Hau], [79Sco], and [84Cen]. Ordering in FeNi after either electron or neutron irradiation was observed by [78Cha] and [79Cha]. FeNi superstructure was observed in meteorites by [77Pet] and investigated further by [79Sco], [80Meh], and [82Gol]. In a study of meteorites, [82Jag] found that compositions of 30 to 40 at.% Ni consisted of ordered $(\gamma Fe,Ni)$ + ordered FeNi, whereas [79Lin], in a STEM study of Fe-Ni meteorites, found decomposition into $(\alpha Fe) + (\gamma Fe,Ni)$ with -5 and 47 at.% Ni, respectively.

[82Gol] proposed a metastable phase diagram, with FeNi having AuCu superlattice structure to account for the "cloudy zone" structure found in meteorites [78Alb1, 78Alb2]. [84Ros1] and [84Ros2] recently proposed that ordered FeNi be included as an equilibrium phase in the Fe-Ni diagram. Figure 6 compares the cluster variation calculations of [84Yam] for the ordering reactions with the assessed diagram of Fig. 1. Also shown in Fig. 10 are the calculations by [85Chu2], [85Chu3], and [86Chu] of a possible tricritical point and spinodal arising from the magnetic interaction. [71Hau] identified Fe3Ni order in Invar alloys using electron diffraction and pointed out its similarity to Fe₃Ni. Computer modeling of the order-disorder transformation in FeNi3 was performed by [74Gol]. Recent work on Fe-Ni meteorites by $[89$ Reu] also pointed to the existence of FeNi and Fe₃Ni in the equilibrium diagram.

The FeNi and Fe₃Ni ordered structures are not shown on the diagram of Fig. 1. Although still an open question, it is probable that these ordered phases are metastable or unstable structures

Table 3 FeNi3 Equilibrium Boundary Measurements

reached in alloys in which the sluggish (γ Fe,Ni) $\rightarrow (\alpha$ Fe) + FeNi₃ eutectoid reaction has been suppressed.

Crystal Structures and Lattice Parameters

A summary of the crystal structures found in Fe-Ni alloys is given in Table 4, and lattice parameters are listed in Table 5. Measured values of the lattice parameters for the (αFe) and $(\gamma Fe,Ni)$ phases vs composition are shown in Fig. 7 and 8. The solid lines in Fig. 7 and 8 represent a weighted least-squares fit to these values.

[37Owe1] found that the (γ Fe,Ni) phase lattice parameter at room temperature (20 "C) as a function of composition reaches a maximum value of 0.3597 nm at 39 at.% Ni and then diminishes at almost the same rate at which it increases. [37Bra] made lattice spacing measurements on alloys containing 27 to 100 at.% Ni that were quenched from 700, 800 and 900 *C. This quenching gave larger lattice spacings than annealed, ordered alloys. Values shown in Fig. 8 are for their annealed alloys, which are more representative of equilibrium. [26Osal] and [26Osa2] measured the lattice parameters and the densities for alloys annealed at 1150 *C and then slow cooled. [63Day] investigated the variation in lattice parameter in FeNi₃ on annealing at 480 $^{\circ}$ C for up to 1000 h.

Lattice parameters were also measured by [21And], [22Kir], [23Bail, [23Mck], [27Jun], [31Phr], [37Owel], [37Owe2], [37Owe3], [37Owe4], [37Owe5], [41Owe], [49Hah], [53Wak], [54Lih], [55Roy], [55Sut], [66Abr], [69Ree], [69Asa], and [83Sen]. The most extensive work is that of [41Owe]. Over most of the region of the Fe-Ni diagram; lattice parameters measured at room temperature will depend on the exact heat treatment given the alloy.

Thermodynamics

[Hultgren,B] assessed the thermodynamic work on Fe-Ni through about 1972. [73Kau] and [70Prel] reviewed thermodynamic properties of the system for use in calculations of the **phase diagram. Since then, phase diagram calculations were**

Table 4 Fe-Ni Crystal Structure Data

Table 5 Fe-Ni Lattice Parameter Data

made by [74Bas], [74Rao], [77Has], [77Lar], [79Lar], [80Lar], [81Imr], [81Nis], [82Cha], [82Vel], [85Chu2], [86Chu1], and $[86Chu2]$.

[74Rao] analyzed phase diagram data, together with thermochemical information, to deduce analytical equations for the thermodynamic functions in solutions. [77Kub] assessed partial and integral enthalpies and entropies of mixing for (yFe,Ni) alloys. [82Vel] derived analytical polynomial expressions for the integral enthalpies of mixing, excess entropies, and the Gibbs energies for the liquid and (yFe,Ni) alloys and used those expressions for the evaluation of the phase diagram.

Enthalpies of mixing of liquid alloys were measured in calorimeters by dropping one pure solid component into a bath with the other component molten. This method was used by [71Toz], [81Igu], [74Bat], and [66Elt]. [70Pre1] used differential thermal analysis for determination of the heat of mixing. The study of [66Elk] contradicts the other investigations, has considerable scatter of experimental values, and was dropped from further consideration. The study of [71Toz] gave unreasonably low values of heats of mixing; they were replaced with new values by the same authors in [81Igu]. The experimental results of [81Igu], [74Bat], and [70Pre1] agree satisfactorily (see Fig. 9).

Enthalpies of mixing for solid alloys were obtained by calorimetric measurement of the heat of reaction between powdered components at 1400 to 1600 K. This method was used by

[63Den], [67Kub], and [71Spe]. [61Ste] dissolved solid components and alloys in liquid tin inside the calorimeter at 1123 K. The results of [63Den], [67Kub], [71Spe], and [61Ste] agree satisfactorily and they are also shown in Fig. 9. When all the experimental results are plotted in one graph (see Fig. 9), it is obvious that the data for solid and liquid alloys are partially overlapping, *i.e.*, the enthalpies of mixing for solid and liquid alloys are the same, within the experimental error.

The problem of differentiating the data for liquid and solid alloys was discussed by [77Kub] and [70Pre1]. [70Pre1] measured heats of melting of ten alloys. Their results deviate from the additive value by 400 to 700 J/mol, which can be compared with the errors in determination of the heat of melting of components given by [70Pre2]: \pm 750 J/mol. If the experimental data for both the solid and liquid heats of mixing are least-squares fitted to a four-term Redlich-Kister expression, the following equation is obtained for the excess enthalpy:

$$
H^{\text{ex}} = X_{\text{Fe}} X_{\text{Ni}} [-16\ 226 - 14\ 982(X_{\text{Fe}} - X_{\text{Ni}}) + 200(X_{\text{Fe}} - X_{\text{Ni}})^2 + 11\ 382(X_{\text{Fe}} - X_{\text{Ni}})^3]
$$

This curve is plotted as a solid line in Fig. 9. The uncertainty in the integral values of heat of mixing is on the order of ± 600 J/mol. In Fig. 9, the dotted line represents the liquid excess enthalpies calculated from the thermodynamic model below, and the dashed line represents the solid excess enthalpies calculated from the same model.

Table 6 Thermodynamic Parameters Used to Modal the Fe-Ni Phase Diagram

Liquid phase

 $G(Fe,L)=0$ $G(Ni,L) = 0$ $G^{ex}(\text{L}) = X_{\text{Fe}} X_{\text{Ni}}[-16\,391 + 3.17\,T + (12\,075 - 2.6\,T)(X_{\text{Fe}} - X_{\text{Ni}}) + (-2000 + T)(X_{\text{Fe}} - X_{\text{Ni}})^2 + (-1500 - T)(X_{\text{Fe}} - X_{\text{Ni}})^3]$ bcc phase $G(\text{Fe},\text{bcc}) = 12\,736.4 - 17.216\,T + 23.18\,T\ln T - 0.0048155\,T^2$ $G(Ni, bcc) = -12500 + 9T$ $G^{ex}(\text{bcc}, T > 1667 \text{ K}) = X_{\text{Fe}} X_{\text{Ni}} [1950 - 3.05 \text{ T} + (-2000 + T)(X_{\text{Fe}} - X_{\text{Ni}})^2 + (-1500 - T)(X_{\text{Fe}} - X_{\text{Ni}})^3]$ $G^{ex}(bcc, T < 1185 K) = X_{Fe} X_{Ni} [13\,274 - 13\,T + (-2000 + T)(X_{Fe} - X_{Ni})^{2} + (-1500 - T)(X_{Fe} - X_{Ni})^{3}]$ $G^{ex}(bcc, 500 < T < 1850 K) = X_{Fe} X_{Ni} (9381 - 8.775 T)$ $G(bcc, mag) = RT_C(bcc) \ln [\beta(bcc) + 1]f(t)$ $t = T/T_C$ (bcc) $f(t < 1) = -0.9053 + t - 0.153t^4 - 0.068t^{10} - 0.00153t^{16}$ $f(t > 1) = -0.06417 t^{-4} - 0.002037 t^{-14} - 0.0004278 t^{-24}$ $T_{\rm C}({\rm bcc}) = 1043X_{\rm Fe} + X_{\rm Fe}X_{\rm Ni}[-757.6 + 1946(X_{\rm Fe} - X_{\rm Ni}) + 2153(X_{\rm Fe} - X_{\rm Ni})^2 - 2779(X_{\rm Fe} - X_{\rm Ni})^3]$ β (bcc) = 2.22X_{Fe} + X_{Fe} X_{Ni} [1.176 + 1.445 (X_{Fe} - X_{Ni}) + 2.275 (X_{Fe} - X_{Ni})² - 2.042 (X_{Fe} - X_{Ni})³]

fee **phase**

 $G(Fe, fcc) = 11\,274 - 16.3878\,T + 22.03\,T \ln T - 0.0041755\,T^2$ $G(Ni,fcc) = 3667.6 - 14.4177 T + 20.113 T \ln T - 0.004561 T^2$ $G^{ex}(fcc) = X_{Fe} X_{Ni}$ [-15 291 + 3.47 T + (12 061 - 2.5 T)($X_{Fe} - X_{Ni}$) + (-2000 + T)($X_{Fe} - X_{Ni}$)² + (-1500 - T)($X_{Fe} - X_{Ni}$)] $G(\text{fcc},\text{mag}) = RT_C(\text{fcc}) \ln [\beta(\text{fcc}) + 1]f(t)$ $t = T/T_C$ (fcc) $f(t < 1) = -0.5597 - 0.6315 t - 0.09178 t⁴ + 0.001872 t¹⁰ - 0.007715 t¹⁶$ $f(t > 1) = -0.03184 t^{-4} + 0.002468 t^{-14} - 0.0019904 t^{-24}$ $T_{\rm C}({\rm fcc}) = -80X_{\rm Fe} + 627.4X_{\rm Ni} + X_{\rm Fe}X_{\rm Ni}$ [2040.5-1250($X_{\rm Fe} - X_{\rm Ni}$)-2627 ($X_{\rm Fe} - X_{\rm Ni}$)²-1784 ($X_{\rm Fe} - X_{\rm Ni}$)³] β (fcc) = -1.59 X_{Fe} + 0.62 X_{Ni} + X_{Fe} X_{Ni} [8.644 + 7.691 (X_{Fe} - X_{Ni}) + 4.435 (X_{Fe} - X_{Ni})² + 0.585 (X_{Fe} - X_{Ni})³]

FeNi3 **phase**

G(Fe in FeNi₃) = 1127.4 - 16.388 T + 22.03 T ln T + 0.0041755 T ² G(Ni in FeNi3) = 3667.6 - 14.418 $T + 20.11$ T ln $T - 0.0045610$ T² $G^{ex}(FeNi_3) = X_{Fe}X_{Ni}[-24185 + 1.9 T + 21475 (X_{Fe} - X_{Ni}) + (-1700 + T) (X_{Fe} - X_{Ni})^2 + (-1500 - T)(X_{Fe} - X_{Ni})^3]$ $G(\text{FeNi}_3,\text{mag}) = RT_C(\text{FeNi}_3) \ln[\beta(\text{FeNi}_3) + 1] f(t)$ $t = T/T_C$ (FeNi₂) $f(t < 1) = -0.5597 - 0.6315t - 0.09718t^4 + 0.001872t^{10} - 0.007715t^{16}$ $f(t > 1) = -0.03184 t^{-4} + 0.002468 t^{-14} - 0.0019904 t^{-24}$ $T_{\rm C}$ (FeNi₃) = -80 $X_{\rm Fe}$ +627.4 $X_{\rm Ni}$ + $X_{\rm Fe} X_{\rm Ni}$ [2040.5 – 1250($X_{\rm Fe} - X_{\rm Ni}$) – 2627 ($X_{\rm Fe} - X_{\rm Ni}$)² – 1784($X_{\rm Fe} - X_{\rm Ni}$)³] β (FeNi₃) = -1.59X_{Fe} + 0.62X_{Ni} + X_{Fe} X_{Ni}[8.644 + 7.691(X_{Fe} - X_{Ni}) + 4.435(X_{Fe} - X_{Ni})² + 0.585(X_{Fe} - X_{Ni})³]

Note: X_{Fe} and X_{Ni} are atomic fractions; Gibbs energy values are in J/mol; and T is in K.

Measurements of the activities in liquid alloys using the transportation method with a stream of inert gas were made by [59Zel] at 1830 to 1891 K, by [78Marl at 1873 K, and by [66Oni] in dilute alloys at 1825 to 1930 K. The transpiration method based on analysis of condensate evaporated in vacuum was used by [59Spe] at 1783 to 1873 K, by [72Mil] at 2178 to 2558 K, and by [71Tse] at 1873 K. The Knudsen cell with a mass spectrometer was used by [77Kub] at 1773 to 1923 K, by [78Con] at 1500 to 1900 K, by [67Bell at 1863 K, and by [81Ram] at 1373 to 1923 K. The results obtained by [71Tse] deviate considerably from other studies and were discarded. All other investigations appear to be in reasonable agreement, at least over most of the concentration range. Most of the results are plotted in Fig. 10 and 11. (In these figures, the dotted lines represent the values calculated from the thermodynamic model given below.) From the analysis of information given in the original papers, it is difficult to reject a study completely or to give preference to any particular group of investigations.

[72Day], [78Con], and [81Ram] also made measurements of activities in the solid alloys. Solid alloy activity measurements using CO/CO2 equilibrium over the Fe-Ni-O system were made by [77Gri] at 1573 K for alloys between 0 and 27 at.% Ni. [76Rob] reported on Ni activities in two alloys with 4.8 and 9.4 at.% Ni at 1400 to 1550 K measured by Knudsen cell with a mass spectrometer. These results agree satisfactorily and are plotted in Fig. 12 and 13. (In these figures, the dotted lines represent the values calculated from the thermodynamic model given below.) Early investigations of solid activities were made by [58Lyu], who measured vapor pressures of alloys using a mass spectrometer at 1463 to 1583 K and by [74Vre] using the transpiration method at 1509 K for alloys between 13 and 94 at.% Ni. The results of [58Lyu] are close to ideal values for both components--in considerable contradiction with the other results-and were dropped from further consideration. The results of [74 Vre], plotted in Fig. 12 and 13, were given low weight because of their scatter and contradiction with the later investigations.

Table 7 Average Magnetic Moments Measured for Ferromagnetic Fe-Ni Alloys

| | Composition, | μ, Bohr | |
|----------------|--------------|-------------------|---------------|
| Phase | at. % Ni | magnetons | Reference |
| $(\alpha$ Fe). | 0 | 2.22 | [63Cra] |
| | 1.4 | 2.21 | $[25$ Pes $]$ |
| | 1.5 | 2.24 | [63Cra] |
| | 3.0 | 2.26 | [63Cra] |
| | 3.5 | 2.22 | $[25$ Pes $]$ |
| | 4.3 | 2.27 | [63Cra] |
| | 5.1 | 2.23 | [25Pes] |
| | 6.2 | 2.29 | [63Cra] |
| | 6.9 | 2.24 | [25Pes] |
| | 9.3 | 2.28 | [63Cra] |
| | 11.3 | 2.23 | [25Pes] |
| | 11.5 | 2.29 | [63Cra] |
| | 15.9 | 2.22 | $[25$ Pes $]$ |
| | 18.9 | 2.23 | [63Cra] |
| | 19.0 | 2.21 | $[25$ Pes $]$ |
| | 23.3 | 2.17 | [63Cra] |
| | 25.2 | 2.13 | $[25$ Pes $]$ |
| | 25.5 | 2.12 | [25Pes] |
| | 29.7 | 2.00 | [25Pes] |
| | 30.4 | 2.00 | [63Cra] |
| | 0 | 2.20 | [25Pes] |
| (YFe,Ni) | 32.5 | 1.93 | [63Cra] |
| | 30.8 | 1.25 | [63Cra] |
| | 32.1 | 1.61 | [63Cra] |
| | 34.3 | 1.77 | [63Cra] |
| | 35.3 | 1.82 | [63Cra] |
| | 40.8 | 1.80 | [25Pes] |
| | | 1.84 | [63Cra] |
| | 42.0 | 1.79 | [25Pes] |
| | 43.5 | 1.77 | $[25$ Pes $]$ |
| | 49.6 | 1.67 | [25Pes] |
| | 49.8 | 1.69 | [63Cra] |
| | 51.0 | 1.65 | [25Pes] |
| | 57.7 | 1.52 | [25Pes] |
| | 60.0 | 1.50 | [63Cra] |
| | 67.0 | 1.36 | [25Pes] |
| | 79.7 | 1.07 | [63Cra] |
| | 80.2 | 1.06 | [25Pes] |
| | 87.7 | 0.88 | [25Pes] |
| | 100 | 0.61 | [25Pes, |
| | | | 63Cra] |

With decreasing temperature, the vapor pressure and the reliability of activity measurements drop rapidly. The difference in values of the Ni activity coefficients at 1473 K (Fig. 12) between two similar investigations [78Con, 67Bel] looks natural, considering the probable uncertainty of the methods used. For both solid and liquid alloys, the uncertainty in determination of the activity coefficient of either of the components is on the order of ± 0.03 for a component in an alloy near the pure component, ± 0.08 in alloys around the equiatomic composition, and ± 0.15 for a component in alloy near infinite dilution.

Other reports of activities in solid alloys were provided by [49Kub], [53Ori], [69Kus], [64Roe], [70Gat], [72Day], [75Tri], [76Dal], [77Ono], and [79Tan]. Most of these contradict each other, and it is worthwhile to plot only some of the latest studies. [75Tri] measured $CO/CO₂$ equilibrium over the Fe-Ni-O system at 1273 K. [76Dal] used the same technique at 1065 to 1380"K. [77Ono] measured the Fe activities by the emf method **at** 1023 to 1423 K. Figures 14 and 15 show that at 1273 K, the results of three of these studies agree satisfactorily. (The dotted lines in the figures represent the values calculated from the thermodynamic model given below.)

Specific heat measurements in the Fe-Ni system at low temperatures were reported by [40Kee], [64Gup], [65Ehr], and [66Shi] and at higher temperatures by [39Lee], [73Kol], and [82Bro2].

An attempt was made to construct a thermodynamic model that represents the Fe-Ni diagram over the widest possible temperature range and agrees, within the above stated experimental errors, with the thermodynamic measurements. This model was used as an aid in the evaluation of the measured phase diagram boundaries, and greater weight was given to these boundaries than to the thermodynamic measurements. In the model, the Gibbs energy for a phase p is represented by:

$$
G(p) = X_{\text{Fe}}G(p,\text{Fe}) + X_{\text{Ni}}G(p,\text{Ni}) + G(p,\text{mag})
$$

+
$$
RT(X_{\text{Fe}} \ln X_{\text{Fe}} + X_{\text{Ni}} \ln X_{\text{Ni}}) + G(p)^{\text{ex}}
$$

where $G(p, Fe)$ is the Gibbs energy of pure Fe, and $G(p)$ ^{ex} is the nonmagnetic excess free energy. Following [74Ind], the magnetic contribution to the Gibbs energy is of the form:

 $G(p, mag) = RT_C(p) \ln (\beta(p) + 1)f(t)$

where t is the reduced temperature $T/T_{\rm C}$, $T_{\rm C}$ is the Curie temperature, and β is magnetic moment in Bohr magnetons. Both $T_{\rm C}$ and β are functions of composition. In the Fe-Ni system, all three phases in the equilibrium diagram are ferromagnetic, and the magnetic contribution is significant, especially at temperatures below 912 "C. Early estimates of the magnetic contributions to phase equilibrium in the Fe-Ni system were given by [73Sch].

The functions for $T_{\rm C}$ and β for the bcc and fcc phases were obtained by least-squares fitting the literature data. For the FeNi3 phase, $T_{\rm C}$ and β values were taken to be approximately equal to those in the fcc phase. For the bcc phase, the function $f(t)$, as expanded in a power series by [78Hil], was used (the resultant contribution to the Gibbs energy agrees well with the alternate form used by [85Chu1]). For the fcc phase, the same power series for $f(t)$ was used, with the coefficients being obtained by leastsquares fitting the evaluated specific heat data of [84Des] for Ni. For pure Fe, the thermodynamic functions given by [79Agr] were used. For pure Ni, the enthalpies of melting and specific heat values given in the evaluation of [84Des] were used to construct the nonmagnetic part of the thermodynamic functions. For bcc Ni, the lattice stability estimates of [73Kau] were used. In assessing the diagram, the thermodynamic modeling and optimization procedure of [81Sun] were used. For the liquidus and solidus, greatest weight was given to the liquidus measurements, especially those of [57Hel].

The parameters found by a combination of optimization and trial and error to best fit the diagram are given in Table 6, and the calculated Fe-Ni diagram is shown in Fig. 16. Above about 500 "C, the calculated and assessed diagrams are nearly identical $(\pm 1^{\circ}C)$ in temperature and ± 0.5 at.% in composition). At lower temperatures, the assessed and calculated boundaries differ somewhat. Especially noticeable is the tricritical point predicted by the model and arising from the influence of the magnetic contribution

| | | Curie. | | | | Curie. | |
|-------|--------------|--------------|----------------------|-------------------|--------------|--------------|----------------------|
| | Composition, | Temperature, | | | Composition, | Temperature, | |
| Phase | at.% Ni | \mathbf{C} | Reference | Phase | at.% Ni | °C | Reference |
| | 0 | 771 | [25Pes] | (γFe,Ni)(cont.) | 55.0 | 558 | [53Wak] |
| | | 770 | [29Gos] | | 57.7 | 590 | $[25$ Pes $]$ |
| | 1.4 | 766 | [25Pes] | | 60.0 | 592 | [53Wak] |
| | 2.8 | 763 | [29Gos] | | | 591 | [63Cra] |
| | 3.5 | 758 | $[25$ Pes $]$ | | 65.0 | 613 | [53Wak] |
| | 4.8 | 755 | [29Gos] | | 67.0 | 612 | $[25$ Pes $]$ |
| | 5.1 | 752 | $[25$ Pes $]$ | | 68.0 | 616 | [53Wak] |
| | 6.9 | 748 | $[25$ Pes $]$ | | 70.0 | 614 | $[53\text{Wak}]$ |
| | 15.9 | 740 | $[25$ Pes $]$ | | 72.0 | 608 | [53Wak] |
| | 19.0 | 715 | $[25$ Pes $]$ | | 74.0 | 600 | [53Wak] |
| | 23.0 | 565 | [43H _{OS}] | | 75.0 | 598 | [53Wak] |
| | 29.7 | 597 | $[25$ Pes $]$ | | 76.0 | 589 | [53Wak] |
| | 31.3 | 535 | $[25$ Pes $]$ | | 78.0 | 585 | [53Wak] |
| | 33.3 | 460 | $[25$ Pes $]$ | | 79.7 | 570 | [63Cra] |
| | 33.8 | 435 | $[25$ Pes $]$ | | 80.0 | 577 | [53Wak] |
| | 29.7 | 120 | $[25$ Pes $]$ | | 80.2 | 576 | [25Pes] |
| | 30.8 | 100 | [63Cra] | | 81.0 | 571 | [53Wak] |
| | 31.3 | 160 | [25Pes] | | 85.0 | 543 | [53Wak] |
| | 31.8 | 174 | [25Pes] | | 87.7 | 511 | $[25$ Pes $]$ |
| | 32.1 | 156 | [63Cra] | | 100.0 | 360 | $[25$ Pes $]$ |
| | 33.3 | 228 | $[25$ Pes] | | | 361 ± 1 | [63Cra] |
| | 33.8 | 245 | $[25$ Pes $]$ | | | 354.3 | [68Kou] |
| | 35.3 | 261 | $[25$ Pes $]$ | FeNi ₃ | 45.0 | 494 ± 5 | [53Wak] |
| | | 228 | [63Cra] | | 50.0 | 543 | [53Wak] |
| | 36.1 | 250 | [63Cra] | | 55.0 | 580 | [53Wak] |
| | 37.0 | 285 | $[25$ Pes $]$ | | 60.0 | 616 | [53Wak] |
| | 38.5 | 300 | [29Gos] | | 65.0 | 636 | $[53 \text{Wak}]$ |
| | 39.0 | 317 | [25Pes] | | 68.0 | 668 | $[53 \text{Wak}]$ |
| | 40.8 | 321 | $[25$ Pes $]$ | | 70.0 | 680 | $[53\text{Wak}]$ |
| | | 354 | [63Cra] | | 72.0 | 696 | $[53\text{Wak}]$ |
| | 42.0 | 346 | $[25$ Pes $]$ | | 74.0 | 691 | $[53 \, \text{Wak}]$ |
| | 43.5 | 403 | $[25$ Pes $]$ | | 75.0 | 681 | $[53 \text{Wak}]$ |
| | 45.0 | 468 | [53Wak] | | 76.0 | 654 | [53Wak] |
| | 47.1 | 415 | [29Gos] | | 78.0 | 624 | [53Wak] |
| | 49.3 | 506 | $[25$ Pes $]$ | | 80.0 | 599 | $[53\text{Wak}]$ |
| | 49.8 | 513 | [63Cra] | | 81.0 | 584 | [53Wak] |
| | 50.0 | 520 | $[53\text{Wak}]$ | | 85.0 | 543 | [53Wak] |
| | 51.0 | 522 | $[25$ Pes $]$ | | | | |

Table 8 Measured Curie Temperatures vs Composition for Fe-Ni Alloys

to the Gibbs energy. (The tricritical point was also calculated by [77Hut].) [85Chu2] gave convincing arguments for the existence of this tricritical point, but further experimental confirmation appears necessary. In fitting the $(\alpha Fe)/(\delta Fe)$ and $(\alpha Fe)/(\gamma Fe, Ni)$ experimental boundaries, better fits are obtained if the expression for the excess energy of the bcc phase is divided into two temperature regions—one for the (α Fe) and one for the (δ Fe) region. An expression for the bcc excess energy that covers the entire temperature range, which gives a good approximation to the observed boundaries and which is more sensible from a thermodynamic point of view, is also included in Table 6.

In Fig. 9, the heats of mixing obtained from the parameters of Table 6 are compared with the measured values (the dotted line is for the liquid, the dashed line for the solid). In Fig. 10 through 15, the theoretical values of the activities calculated from the model parameters (dotted lines) are compared with measured values. In all instances, agreement is within experimental error. However, the fits are in some respects unsatisfying, especially for the Nirich heats of mixing and Fe-rich Ni activities. The slight oscillation in the Ni activity coefficients on the Fe-rich side (Fig. 12 and 14) evidently is unjustified by the data. This may be in part due to

the poor accuracy of the measured values of the solidus and liquidus of the Ni rich alloys.

Pressure

The equilibrium diagram for 50,100, and 150 kbar was calculated by [61 Kau 1]. [61 Kau 2] showed that pressure lowers the $(\gamma Fe, Ni)$ $\rightarrow (\alpha \text{Fe})$ transformation temperatures. [66Mcq] investigated the effect of pressure on the density of Fe-Ni alloys by the shock technique up to 2000 kbar. Using XRD, [68Tak] measured the effect of pressure on the crystal structure and molar volume.

Magnetism

The saturation magnetization, average magnetic moments, and Curie temperatures for single-phase foe alloys from 30 to 100 at.% Ni were measured by [63Cra]. They also measured the average magnetic moment for single-phase bee alloys in the range 0 to 30 at.% Ni. Magnetic moments on individual Fe and Ni atoms were measured by [62Col], [73Cab], [74Nis], and [55Shu] between 40 and 100 at.% Ni. The use of magnetic measurements for

studying two-phase Fe-Ni alloys was described by [39Suc] and [40Pie]. The effect of heat treatment on the magnetic properties of FeNi₃, including the maximum permeability, coercive force, remanent, and saturation magnetization was investigated by [53Wak]. By extrapolating the magnetization vs temperature curves, they estimated the Curie temperatures for ordered FeNi and found a higher Curie temperature for the ordered alloys than for the disordered. A slightly higher saturation magnetization is also found for the ordered alloys ([40Gra], see Table 7). Figure 17 and Table 7 compare the various measured magnetic moments, and Fig. 18 and Table 8 compare the various measured Curie temperatures. The Curie temperature of pure Ni is taken as 627.4 K, in accordance with [68Kou] and [82Rhy].

Alloys of high maximum permeability, the so-called permalloys, can be formed by rapidly cooling alloys near the FeNi₃ composition [32Dah, 38Kay, 53Boz1, 53Boz2, 64Sch]. The magnetic properties of an equiatomic single crystal of FeNi that had been ordered metastably in the AuCu structure by neutron irradiation below 320 *C were studied by [64Nee]. Further anomalies in magnetic properties and phase separation have been observed by [51Suc], [59Dek] and [61Gor]. [75Ind] showed how the Curie temperature variation in (γ Fe,Ni) may be closely fitted to a Redlich-Kister form with a single interaction term. [77Mio] showed how this interaction parameter is related to the two-moment model for γ Fe of [63Wei]. [73Miz] measured Curie temperatures and magnetic moments in rapidly quenched amorphous quasibinary alloys with 0 to 90 at.% Ni and boron and phosphorus additions.

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