The Nickel-Rich Region of the Ni-Ge Phase Diagram

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We present new results on the phase boundaries separating the single and the 2-phase regions of the Ni-rich Ni-Ge phase diagram. The accuracy of the boundary between the 2-phase Ni(Ge) + Ni₃Ge and single-phase Ni₃Ge region is reinforced by the addition of new data obtained from dissolution experiments. This boundary is consistent with the results of a recent thermodynamic model at temperatures lower than ~840 °C, but is leaner in Ge content at higher temperatures. Additional data on the equilibrium solubility of Ge in Ni (Ni₃Ge solvus) reinforces the accuracy of the phase boundary to temperatures as low as 700 °C; these data are not new, but are not included in the most recent assessments. The recent thermodynamic model of the Ni₃Ge solvus is in remarkably good agreement with the experimental results. The composition dependence of the ferromagnetic Curie temperature, T_c , is also re-evaluated by the addition of data not previously included in recent assessments. The dependency of T_c on Ge content is essentially linear to concentrations of ~13% Ge, which exceeds the solubility of Ge in Ni-Ge alloys. Empirical equations relating equilibrium compositions and temperatures of the phase boundaries and the composition dependence of T_c are presented.

Keywords equilibrium diagram, experimental phase equilibria, metallic alloys, phase diagram, phase equilibria, precipitation, solubility limit

1. Introduction

In the most recent version of the Ni-rich region of the Ni-Ge phase diagram Ikeda et al.^[1] have shown that the equilibrium composition of the [Ni(Ge) + Ni₃Ge]/Ni₃Ge phase boundary decreases with increasing concentration of Ge. This indicates that precipitation of the disordered Ni-Ge solid solution from supersaturated hypostoichiometric Ni₃Ge should be amenable to investigation. The retrograde nature of this phase boundary is also consistent with the observation^[2] of disordered Ni-Ge precipitates in very large Ni₃Ge particles observed in samples that were slowly cooled from a high aging temperature. These factors leave no doubt that supersaturated Ni₃Ge alloys belong to a very small class of so-called “inverse” alloys, of which Ni₃Al is a member.^[3–6]

We reported preliminary work on the decomposition of supersaturated, hypostoichiometric Ni₃Ge several years ago.^[7] The need for accurate data on the phase boundaries to calculate the volume fractions of the Ni-Ge solid-solution precipitates in aged inverse Ni₃Ge alloys stimulated this investigation. Additionally, since studies of precipitation are often conducted at lower temperatures than those usually amenable to experimental phase diagram determinations, we also needed to re-evaluate the limits of solubility of the

Ni-Ge solid solution (the γ' solvus). Finally, thermodynamic modeling of Ni-rich Ni-base alloys generally involves a magnetic contribution to the Gibbs free energy of mixing,^[8,9] even at temperatures above the ferromagnetic Curie temperature, T_c . For this reason we also include all known data in a re-evaluation of the dependence of T_c on composition.

To make the identification of the phase boundaries less cumbersome, we call the Ni-Ge solid solution the γ phase and the Ni₃Ge phase the γ' phase, by analogy with the Ni-Al alloy system. The γ phase is face-centered cubic and the γ' phase, like Ni₃Al, has the ordered L1₂ crystal structure. We call the $\gamma/(\gamma + \gamma')$ phase boundary the γ' solvus, which is standard nomenclature. To distinguish this phase boundary from the $(\gamma + \gamma')/\gamma'$ phase boundary, we refer to it as the γ solvus.

2. Experimental Details

Binary Ni-Ge alloys containing 22.0, 22.5, and 23.0 at.% Ge were purchased from the Ames Laboratory, where they were annealed in vacuum at 1000 °C for 100 h prior to shipping.¹ The alloys were received in the form of rods 10 mm in diameter, weighing $\sim 30 \pm 0.6$ g. The reported weight losses were 0.073, 0.033, and 0.066 wt.%, respectively. An alloy containing 23.5% Ge, weighing ~29.5 g, was prepared in our laboratory by arc melting 99.995% Ni and 99.9999% Ge. This alloy was melted 3 times and annealed in vacuum at 1000 °C for 100 h. The total weight loss was about 0.133 wt.%, approximately 0.033 wt.% occurring during vacuum annealing. Since the weight losses in our arc-melted alloy could not be preferentially attributed to either Ge or Ni, the composition is taken as 23.5% Ge.

¹Unless indicated otherwise, the units of concentration in the alloys are either in atomic percent or atom fraction.

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The as-received samples were metallographically polished, etched in a solution consisting of 50% HNO_3 , 25% CH_3COOH , and 25% H_2O for about 10 s and examined optically using a Nikon Eclipse Me600 microscope. Optical micrographs of the alloys purchased from the Ames Laboratory are shown in Fig. 1. The microstructures of the alloys containing 22.0 and 22.5% Ge were clearly dendritic, as seen in Fig. 1(a) and (b), whereas the alloy containing 23.0% Ge was single phase after the same annealing treatment, as seen in Fig. 1(c). The alloy containing 23.5% Ge was single phase after annealing. Energy-dispersive X-ray spectroscopy was performed using a Cambridge Stereoscan 250 scanning electron microscope operating at 20 keV to determine the chemistry of the Ni_3Ge phase; the known compositions of the alloys were used as calibration standards. The compositions of the γ' phase in the two alloys nominally containing 22.0 and 22.5% Ge were found to be $22.48 \pm 0.24\%$ and $22.55 \pm 0.20\%$, respectively.

Slices were cut from the cast alloys by electric-spark discharge machining, solution-treated in an inert protective argon atmosphere at $1000 \pm 1^\circ\text{C}$ for 0.75 h and quenched into refrigerated brine. All the heat-treatments described in this work were done on the slices. Disks 3 mm in diameter were mechanically punched from the slices and mechanically ground by hand to thicknesses of $\sim 150\ \mu\text{m}$ using #600 carbide emery paper. The discs were electrochemically dimpled using a solution of 20% HNO_3 , 20% H_3PO_4 , 15% CH_3COOH , and 45% H_2O at 90 V, then thinned to perforation at 20 V using a solution of 10% HClO_4 and 90% $\text{C}_2\text{H}_5\text{OH}$ maintained at -10 to 0°C . Electron microscopy was performed using a JEOL 100 CX TEM-SCAN at an operating voltage of 100 kV. The precipitate microstructures were best viewed using dark field images taken with a γ' (100) superlattice reflection from thin foils in [001] orientation, so the precipitates generally appear dark against a bright background.

3. Ni-Ge Phase Diagram

3.1 The γ Solvus

To establish the $(\gamma + \gamma')/\gamma'$ phase boundary as accurately as possible we used dissolution experiments similar to those for establishing the γ solvus in the Ni-Al alloy system.^[4] Rastogi and Ardell^[10] used the same method to help determine the coherent solvus curves for γ' -type precipitates in Ni-Al, Ni-Si, and Ni-Ti alloys. This method involves the initial aging of specimens in the 2-phase region of the phase diagram for the purpose of producing copious quantities of precipitates. The specimens are then re-heated for short times, typically an hour, at temperatures near the solubility limit, then quenched and examined by TEM. There is no barrier to dissolution of the precipitates, so their absence on re-heating indicates that the specimens were re-heated in the single-phase region of the phase diagram. The accuracy of the method is limited by the temperature interval chosen for the re-heating experiments. In this work the intervals used were $15\text{--}20^\circ\text{C}$.

The procedure is exemplified by the results shown in Fig. 2. A specimen containing 23.0% Ge was aged initially at 600°C for 24 h in the 2-phase region of the phase diagram to produce large amounts of small γ precipitates. On re-aging at 870°C for 1 h, much larger precipitates are still clearly visible, see Fig. 2(a). On increasing the temperature to 885°C and re-aging for 1 h, the number of precipitates decreased, but their size was larger, as shown in Fig. 2(b). On re-aging another specimen for 1 h at 900°C , it is evident that the precipitates completely dissolved, as seen in Fig. 2(c). This places the temperature of the γ solvus for the alloy containing 23.0% Ge between 885 and 900°C . For the alloys containing 22.55 and 23.50% Ge, the temperatures were found to lie between 970 and 985°C and 810 and 830°C , respectively. Dissolution experiments were not performed on the alloy containing 22.48% Ge.

Fig. 1 Microstructures observed in the Ni-Ge alloys after annealing at 1000°C for 20 h: (a) 22.0% Ge; (b) 22.5% Ge; (c) 23.0% Ge

Fig. 2 The microstructures in the alloy containing 23.0% Ge after solution-treating and aging under the following conditions: (a) 600°C for 24 h + 870°C for 1 h; (b) 600°C for 24 h + 885°C for 1 h; and (c) 600°C for 24 h + 900°C for 1 h

The results of the dissolution experiments are shown in Fig. 3, which also includes the data of Ikeda et al.^[1] It is evident that the two sets of data are in excellent agreement. The best estimate of the $\gamma/(\gamma + \gamma')$ phase boundary is given by the empirical equation

$$T_{\gamma} = \left(1.338243 - 17.282026X_{\text{Ge}|\gamma} + 74.494182X_{\text{Ge}|\gamma}^2 - 107.111771X_{\text{Ge}|\gamma}^3 \right) \times 10^6 \quad (\text{Eq 1})$$

where $X_{\text{Ge}|\gamma}$ is the composition of the γ solvus in atom fraction and T_{γ} is in °C. The data used to fit this curve were the temperatures halfway between those for which Ni-Ge precipitates were seen or not, so the typical uncertainty is ± 7.5 to ± 10 °C. Equation 1 represents the γ solvus quite accurately over the temperature range 700-1000 °C, as indicated by the solid curve in Fig. 3. The excessive number of decimal places seems like an extravagance, but if the numbers are rounded off to as little as four significant figures, as was the case in a recent publication,^[11] the γ solvus temperatures are underestimated by 21-27 °C.

On taking the error bars reported by Ikeda et al. into account, as well as the uncertainty in temperature in our work, it is clearly possible to describe the γ solvus as a linear fit between $X_{\text{Ge}|\gamma}$ and T_{γ} . In this case, however, the equilibrium composition of the γ' phase would exceed 0.25 at temperatures below about 545 °C. This is inconsistent with expected compositions of the Ni_3Ge phase, hence Eq 1 for the γ solvus is preferred.

Fig. 3 The Ni(Ge) solvus, as indicated by the solid curve calculated using Eq 1. Plotting symbols: $\boxtimes$ 2 phase on re-aging; $\oplus$ single phase on re-aging; $\circ$ Ikeda et al.^[1]; $\bullet$ this investigation

3.2 The γ' Solvus

The data on the solubility limits of the Ni_3Ge phase reported by Ikeda et al.^[1] include the measurements of Dayer and Feschotte^[12] and Komai et al.,^[13] plus the results of their own work. There are additional data on the γ' solvus, some of which^[14,15] were included in a previous compilation^[16] illustrating how data from measurements of the kinetics of solute depletion during particle coarsening can be used to determine equilibrium solubility limits. Here, we consider this method further in light of a new theory of coarsening, involving diffusion of solute through, rather than to, the diffuse interface between γ' -type precipitates and the matrix.

The kinetics of solute depletion during particle coarsening is controlled by the equation

$$X - X_e = (\kappa t)^{-1/n}, \quad (\text{Eq 2})$$

where X is the solute concentration in the matrix at time t , X_e is its thermodynamic equilibrium value, κ is a rate constant, and n is a temporal kinetic exponent. In the familiar theory of diffusion-controlled coarsening of Lifshitz and Slyozov^[17] and Wagner^[18] (the LSW theory), $n = 3$. When the kinetics of coarsening is controlled by diffusion through the interface between the precipitate and matrix phases the temporal exponent can differ from Eq 3, as discussed by Ardell and Ozolins^[19] in the context of their theory of trans-interface-diffusion-controlled coarsening (the TIDC theory). It has been argued that n can take on different values depending on the relationship between the particle size and interface width, and shown that n can be determined from the particle size distribution.^[19,20] The TIDC theory has been used recently^[11] to re-evaluate data on coarsening to extract new values of the γ/γ' interfacial free energies in Ni-Al, Ni-Ga, Ni-Ge, Ni-Si, and Ni-Ti alloys, in light of thermodynamic models of the Ni-rich solid solutions these systems.

For purposes of this work, we report values of $X_{\text{Ge}|\gamma'}$ [= X_e in Eq 2] obtained from data on the kinetics of solute depletion in Ni-Ge alloys.^[2,15] These data were originally analyzed using $n = 3$, yielding the results reported in the second column of Table 1. These same two sets of data were recently re-analyzed using $n = 2.385$,^[11] producing the values of $X_{\text{Ge}|\gamma'}$ reported in the third column of Table 1. The relevant plots of the data are contained in the references cited, so there is no need to show them again here. It is evident that there is very little difference between the solubility limits resulting from the two different methods of analysis, $X_{\text{Ge}|\gamma'}$ being only slightly larger using the TIDC theory, i.e., Eq 3 with $n = 2.385$.

The Ni-rich region of the Ni-Ge phase diagram is shown in Fig. 4. The results derived from the TIDC analysis (third column of Table 1) are included in the compilation, as are all the data generated since the work of Dayer and Feschotte was published.^[12] The γ' solvus is represented by Eq 3. The γ' solvus was fit to all the data except for the one datum of Brahman et al.^[14] on the solubility at 1100 °C. The equation describing this curve is

$$X_{\text{Ge}|\gamma'} = 0.07191 \exp\{6.9771 \times 10^{-4} T_{\gamma'}\}, \quad (\text{Eq 3})$$

where $X_{\text{Ge}\gamma'}$ is the solvus composition and $T_{\gamma'}$ is the solvus temperature in °C. This equation differs slightly from the one previously published^[11] owing to the addition of the new data. The agreement between the thermodynamic model of Liu et al.^[21] and the γ' solvus is exceptional, Eq 3 describing the results of the model quite accurately.

Data on the γ' solvus reported by Komai et al.^[13] are included in Fig. 4. The concentrations measured by them are generally larger than those in Fig. 3, and for this reason were not used in the determination of Eq 1. It should also be noted that Dayer and Feschotte^[12] reported constant values of $X_{\text{Ge}\gamma'} \approx 0.225$ for the γ solvus over the temperature range 700–1000 °C. Their data clearly disagree with all the other measurements and are not shown. The dashed curve in Fig. 4 represents the results of the model of Liu et al.^[21] The agreement with experiment is very good at temperatures below about 840 °C. However, the model overestimates $X_{\text{Ge}\gamma'}$ at higher temperatures and does not correctly capture the curvature of the γ solvus.

Table 1 Values of $X_{\text{Ge}\gamma'}$ obtained from the analyses of data on solute depletion during coarsening of γ' precipitates in binary Ni-Ge alloys

T , °C	$X_{\text{Ge}\gamma'}$	
	LSW ($n = 3$)	TIDC ($n = 2.385$)
700	0.1171 ± 0.0004	0.1175 ± 0.0004
724	0.1195 ± 0.0001	0.1197 ± 0.0001

Fig. 4 The Ni-rich region of the Ni-Ge phase diagram. The solid curves represent the phase boundaries determined in this work. The dashed curve is the phase boundary predicted by the thermodynamic model of Liu et al.^[21] Plotting symbols: ● this investigation; ○ Ikeda et al.^[1]; ▼ Kim and Ardell^[2]; ◇ Dayer and Feschotte^[12]; □ Komai et al.^[13]; × Brahman et al.^[14]; ▲ Wimmel and Ardell^[15]

3.3 The Composition Dependence of the Ferromagnetic Curie Temperature

For the sake of completeness we present data on the variation of the ferromagnetic Curie temperature, T_c , with X_{Ge} . Liu et al.^[21] evidently included in their model only the data of Ilonca,^[22] which is limited to the stable Ni-Ge solid solution. However, measurements of T_c exist up to about $X_{\text{Ge}} \approx 0.13$.^[15,23] These results are included in this evaluation and shown in Fig. 5. The dependence of T_c on X_{Ge} is linear to a good approximation, and is given by the equation

$$T_c = 355.44 - 4090X_{\text{Ge}}, \quad (\text{Eq 4})$$

which is shown in Fig. 5 as the solid line. The Curie temperature of pure Ni predicted by this equation ($X_{\text{Ge}} = 0$) is within a few °C of the most recent values reported in the literature, namely 355.32 ± 0.74 °C^[24] and 358.28 ± 0.04 °C.^[25] The equation for T_c predicted by the thermodynamic model of Liu et al.^[21] is

$$T_c = (1 - X_{\text{Ge}})T_{c,\text{Ni}} - 3750(1 - X_{\text{Ge}})X_{\text{Ge}}, \quad (\text{Eq 5})$$

where $T_{c,\text{Ni}}$ is the Curie temperature of pure Ni. Rearranging the terms in (5) leads to the equation

$$T_c = T_{c,\text{Ni}} - (T_{c,\text{Ni}} + 3750)X_{\text{Ge}} + 3750X_{\text{Ge}}^2. \quad (\text{Eq 6})$$

If the quadratic term in X_{Ge} is ignored and $T_{c,\text{Ni}}$ is taken as 355.32, the difference between the predictions of Eq 4 and 6 is insignificant, but due to the presence of the quadratic term Eq 6 seriously overestimates T_c at higher concentrations. Specifically, at $X_{\text{Ge}} = 0.10$ and 0.13, T_c

Fig. 5 The variation of ferromagnetic Curie temperature, T_c , with Ge concentration, X_{Ge} , in Ni-Ge solid solutions. Plotting symbols: ● Wimmel and Ardell^[15]; □ Ilonca^[22]; ◇ Lecocq^[23]

is overestimated by ~ 36 and 61 °C, respectively. These differences could affect the magnetic contributions to the Gibbs free energy of mixing under certain circumstances.

4. Summary

New results on the γ solvus are presented. They are in excellent agreement with the data published by Ikeda et al.^[1] The new data also confirm the status of off-stoichiometric Ni_3Ge alloys as inverse alloys, enabling investigation of the precipitation of the disordered Ni-Ge solid solution. The thermodynamic model of Liu et al.^[21] overestimates the concentrations of this phase boundary at temperatures higher than ~ 840 °C, but is in reasonable agreement with the predictions of Eq 1 at lower temperatures. The γ' solvus has been re-evaluated to include all extant data published from the work of Dayer and Feschotte^[12] to the present day. The addition of the new data did not produce any significant changes in the γ' solvus curve published by Ikeda et al.,^[1] but the data improve the reliability of the γ' solvus at lower temperatures. The predictions of the thermodynamic model of Liu et al.^[21] are in remarkably good agreement with these results over the entire range of temperatures. The composition dependence of the ferromagnetic Curie temperature, T_c , is linear up to about 13% Ge. The experimentally measured values of T_c are significantly lower than those predicted by the model of Liu et al. at concentrations between 10 and 13% Ge. Empirical equations are presented for the phase boundaries and ferromagnetic Curie temperature.

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