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Phase Equilibria and Phase Transformation of Co-Ni-Ga Ferromagnetic Shape Memory Alloy System

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Phase equilibria and martensitic and magnetic phase transformations of the β phase in the Co-Ni-Ga system have been investigated. It has been shown that the β phase is in equilibrium with the α-phase over a wide range compositions at 600-1100 °C. The β phase exhibits both a paramagnetic-ferromagnetic transition and a thermoelastic martensitic transition from B2 to $L1_0$ structure. The Curie temperature T_C increases with decreases in both Ni and Ga content, and the martensitic transition starting temperature M_s decreases with decreasing Ni content and with increasing Ga content. The composition region of the β phase exhibiting the thermoelatstic martensitic transformation from ferromagnetic austenite is located near the α + β two-phase region. T_C and M_s of α + β two-phase alloys increase with increasing annealing temperature.

Keywords annealing, ferromagnetic, magnetism, martensite, shape memory

1. Introduction

Ferromagnetic shape memory alloys have received much attention as new sensor and actuator materials since Ullakko et al. [1996Ull] reported a large magnetic field-induced strain by a rearrangement of twin variants in Ni_2MnGa alloy. Several candidates for ferromagnetic shape memory alloys have been reported, including Fe-Pd [1998Jam], Fe-Pt [2000Kak], and Co-Ni-Ga systems [2001Oik2] [2001Wut]. The present authors have conducted systematic studies of the martensitic and magnetic transitions in CoNi-Al [2001Oik1] [2001Oik2], Co-Ni-Ga [2001Oik2], Ni-Fe-Ga [2002Oik1] [2002Oik2], Ni-Fe-Al [2003Oik1], Ni-Mn-Al [1999Gej], and Cu-Mn-Ga [2004Oik] body-centered-cubic (bcc) ordered alloy systems and have found the composition region exhibiting the thermoelastic martensitic transition from the ferromagnetic austenite state.

Co-Ni-Ga β (bcc ordered structure) alloys were explored as candidates for ferromagnetic shape memory alloys [2001Oik2] [2001Wut]. The characterization of the martensitic transition in the Co-Ni-Ga β phase alloy system is suggested to be from B2 to the tetragonal $L1_0$ structure and [2001Oik2] or Heusler $L2_1$ to the tetragonal or orthorhombic structure [2002Cra][2003Kis1][2003Kis2]. Chernenko et al. [2004Che] reported the structure and thermal and mechanical characterization of a Co-Ni-Ga single crystal. They suggested the martensitic transition was probably from $L2_1$ to the tetragonal $L1_0$ structure, although they could not observe the ordered reflections that characterize the $L2_1$ structure. They also showed the stress-induced martensitic transition behavior and superelasticity effect. Li et al. [2004Li] observed the two-way shape memory effect of Co-Ni-Ga single crystals under a magnetic field. Sato et al. [2004Sat] investigated the magnetic properties including the magnetostriction of the Co-Ni-Ga ribbon sample prepared by an electromagnetically controlled melt-spining apparatus. Only limited information exists regarding the phase equilibria of the Co-Ni-Ga system. Chen and Dodd [1985Che] investigated the crystal structure of alloys close to $(Ni,Co)_{50}Ga_{50}$. They reported that the β solid solution phase with the B2 structure is formed from the Co-Ga side to the Ni-Ga side and is in equilibrium with α (A1:fcc disordered structure) phase above 900 °C at the composition of Co 30 at.% Ni-40 at.% Ga as confirmed by x-ray diffraction (XRD) analysis. Mikula et al. [1987Mik] also reported the B2 structure at 900 °C in several Co-Ni-Ga β phase alloys by XRD analysis. Booth et al. [1978Boo] conducted XRD and neutron diffraction analyses of several Co-Ni-Ga β single phase alloys and α + β two-phase alloys annealed at 550 and 830 °C. They concluded that the crystal structure of β phase is the B2 structure and that Ni atoms occupy the Co sites of CoGa with displaced Co atoms moving to the Ga sublattice. In previous work [2001Oik2], phase equilibria between the β and α-phases at 1000 and 1100 °C and the martensitic and magnetic transition temperatures in 30 at.% Ga section were reported.

Although Co-Ni-Ga β alloys are brittle, the ductility can be dramatically improved by the introduction of α-phase [2001Oik2]. Therefore, the α + β two-phase alloys are attractive as candidates for ductile ferromagnetic shape memory alloys. The martensitic and magnetic transition temperatures are sensitive to chemical composition, and

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those of α + β two-phase alloys are expected to depend on the annealing temperature in analogy with the Co-Ni-Al system [2001Oik1]. The current study reports the results of the systematic investigation of the phase stability in the Co-Ni-Ga system including the martensitic, magnetic transitions, and phase equilibria as well as crystal structures and magnetic properties.

2. Experimental

Co-Ni-Ga alloys with compositions in the range Co-(18-50) at.% Ni-(30, 32) at.% Ga were prepared as buttons in 20 g quantities, first by arc melting pure cobalt (99.9%), nickel (99.9%), and gallium (99.999%) under an argon atmosphere, remelting in a cold crucible levitation melting furnace, and casting into a copper mold.

Small specimens were taken from the ingots and sealed in a quartz capsule filled with argon gas. Solution heat treatments at 1200 °C for 4 h and at 1150 °C for one day were conducted to obtain β single-phase samples for alloys containing 30 at.% Ga and 32 at.% Ga, respectively. Heat treatments for equilibration in the two-phase region were carried out at 600 and 800 °C for 30 days, 900 and 1000 °C for two days, and 1100 °C for one day. After the heat treatment, the samples were quenched into ice water. Some alloys were aged at 300 °C to investigate the effect of low-temperature aging on the martensitic transition temperature and the crystal structure.

The microstructure of samples, etched in 7% ferric chloride-18% hydrochloric acid-water solution, was examined by optical microscopy. The chemical compositions of α and β phases in two-phase samples were determined by energy dispersion x-ray spectroscopy (EDX) using a standard calibration method. The phase identification and characterization were carried out by x-ray powder diffraction (XRD) using Fe K_α radiation and transmission electron microscopy (TEM). For the XRD examination, a powder sample ground from the solution-treated sample was sealed in a quartz capsule filled with argon gas. After a heat treatment at 1200 °C for 15 min, the quartz capsule was dropped into ice water. Thin foils for TEM observation were prepared by jet-polishing with 20% perchloric acid-methanol solution.

The Curie temperature T_C and the magnetization I were measured using a vibrating sample magnetometer (VSM), and the martensitic transition temperatures were determined by differential scanning calorimetry (DSC) at cooling and heating rates of 10 °C/min. T_C was defined as the minimum point of the temperature derivative of magnetization in a field of 500 Oe because no straight parallel lines of the Arrot plots could be obtained. This method gives a reliable T_C [1999Sai].

3. Results and Discussion

3.1 Microstructures and β/α Phase Equilibrium

Optical micrographs of Co-25 at.% Ni-30 at.% Ga alloy annealed at 1200 and 900 °C are shown in Fig. 1. A typical twinned martensitic structure is observed in both samples.

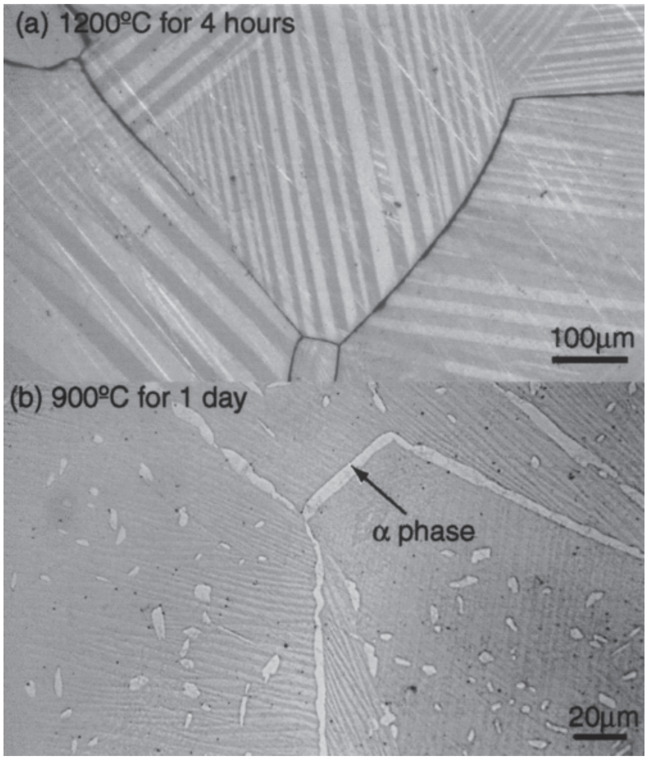

Fig. 1 Optical micrographs of Co-25at.%Ni-30at.%Ga alloy annealed at (a) 1200 °C and (b) 900 °C

The precipitation of a secondary phase is observed in the β matrix in the alloy annealed at 900 °C as shown in Fig. 1(b), although the alloy annealed at 1200 °C exhibits only the β single-phase. The precipitation phase was identified as the α-phase.

Equilibrium compositions of the β and α-phases obtained from the two-phase alloys are listed in Table 1 and are plotted in Fig. 2. The α + β two-phase region extends widely from the Co-Ga side to near the Ni-Ga side, which is very similar to the α/β equilibrium in the Co-Ni-Al ternary system [1996Kai] [2001Oik2]. The composition of Co_2NiGa is located in the α + β two-phase region, namely the Co_2NiGa Heusler stoichiometric compound does not exist in a stable state. It can also be seen that a tie-line direction of phase equilibrium in Co-50at.%Ni-30at.%Ga alloy at 600 °C is considerably different from those of other alloys. This fact suggests that the precipitation phase changes from the α-phase to another phase, which is presumed to be the α′ ($L1_2$ structure) phase in analogy with the Ni-Ga binary alloy phase diagram [1990Mas].

3.2 Martensitic and Magnetic Transitions of β Phase

Figure 3 shows typical DSC and thermomagnetization curves. The endothermic and exothermic reactions corresponding to the martensitic transitions are observed upon heating and cooling, respectively. Temperatures characterized as the martensitic and reverse transitions (M_s, martensitic transition starting temperature; M_f, martensitic transi-

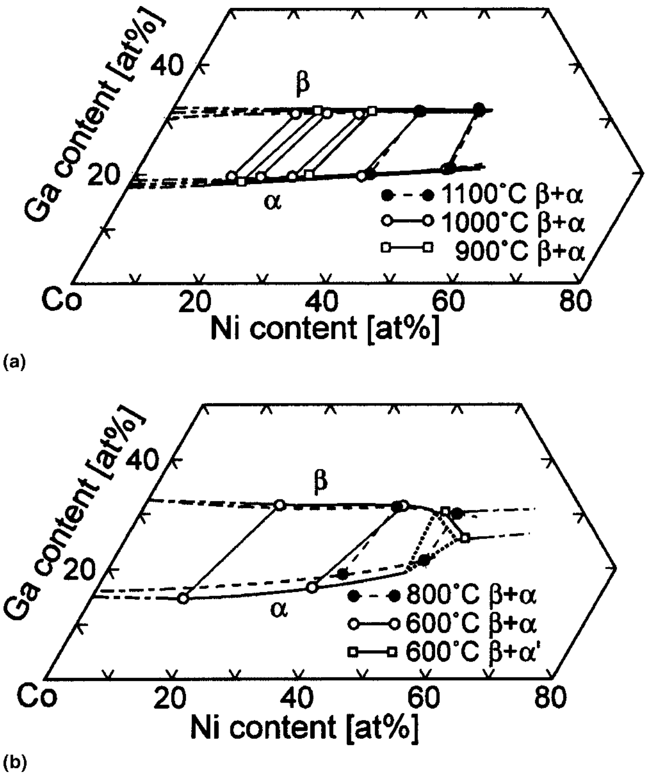

Fig. 2 Phase equilibria at some temperatures from 600 to 1100 °C in Co-Ni-Ga system

Table 1 Composition of β, α, and α′ phases in equilibrium at different temperatures in the Co-Ni-Ga system

Temperature, °C	β phase		α phase	
	Ni, at.%	Ga, at.%	Ni, at.%	Ga, at.%
1100(a)	39.44	31.09	37.52	19.74
	49.01	31.11	49.87	20.82
1000(a)	20.40	30.72	15.98	19.17
	25.22	30.77	20.72	19.11
	30.34	30.58	25.45	19.21
	39.84	31.06	36.59	10.10
	48.91	31.60	48.93	20.67
900	23.55	31.07	18.11	18.44
	32.55	31.08	27.72	19.67
800	39.85	31.02	37.36	18.94
	50.08	29.82	49.11	21.41
600	21.25	31.29	14.74	14.52
	40.98	31.31	34.93	16.35
600	48.31	29.92	53.76(b)	25.20(b)

(a) Data from [2001Oik2]. (b) Data shown are for the α′ phase.

tion finishing temperature; A_s, reverse transition starting temperature; and A_f, reverse transition finishing temperature) were defined as the cross points of the baseline and the tangent of the maximum or minimum inclination in the DSC curves, as shown in Fig. 3(a). The thermomagnetization curves of a Co-22.5at.%Ni-30at.%Ga alloy in a magnetic field of 500 Oe show steps corresponding to the martensitic and reverse transition temperatures. This is because the magnetization saturation is more easily accomplished in the austenite phase than in the martensite phase. This result is similar to those obtained in other ferromagnetic shape memory alloys [2001Oik1] [2002Oik2] [2004Oik].

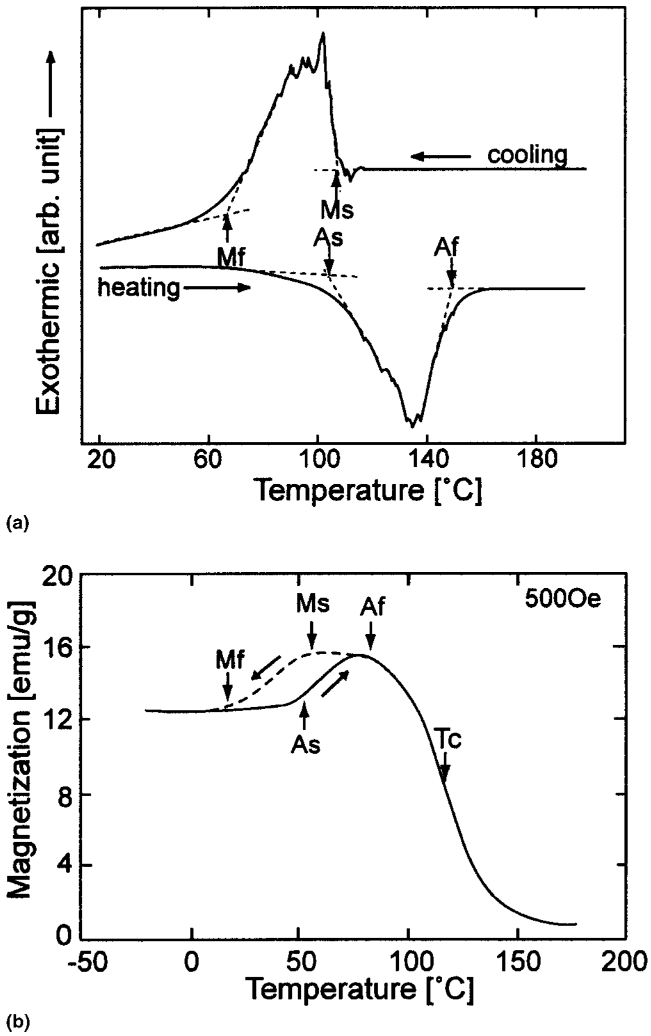

Fig. 3 (a) DSC curve of Co-23.5at.%Ni-30at.%Ga and (b) magnetization-temperature curve of Co-22.5at.%Ni-30at.%Ga quenched from 1200 °C

The M_s, A_f, and T_C of β single-phase alloys obtained by the solution heat treatment are plotted as a function of Ni content in the 32 at.% Ga section in Fig. 4. The value of T_C decreases, and those of M_s and A_f increase with increasing Ni content. The T_C and M_s curves cross at around 24 at.% Ni. It is very interesting to note that the T_C curve in the martensitic phase is not continuous with that in the parent phase and that these curves show a temperature step around the cross point of the M_s and the T_C curves, because T_C of martensite phase is different from that of austenite phase as mentioned in the authors previous paper [2001Oik2]. This transformation behavior is very similar to that of the

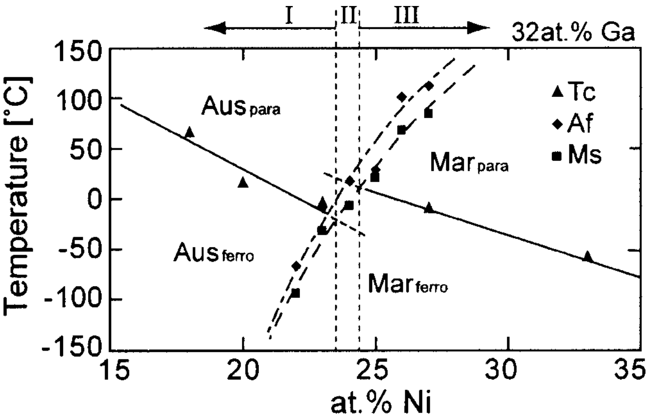

Fig. 4 Composition dependence of T_C, M_s, and A_f of the β single-phase in the Co-Ni-32 at.% Ga section

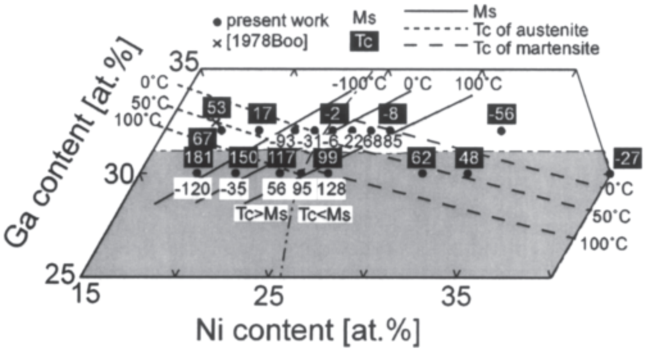

Fig. 5 Composition dependence of T_C and M_s of β single-phase alloys in the Co-Ni-Ga ternary systems; the hatched area is the α + β two-phase region at 1100 °C.

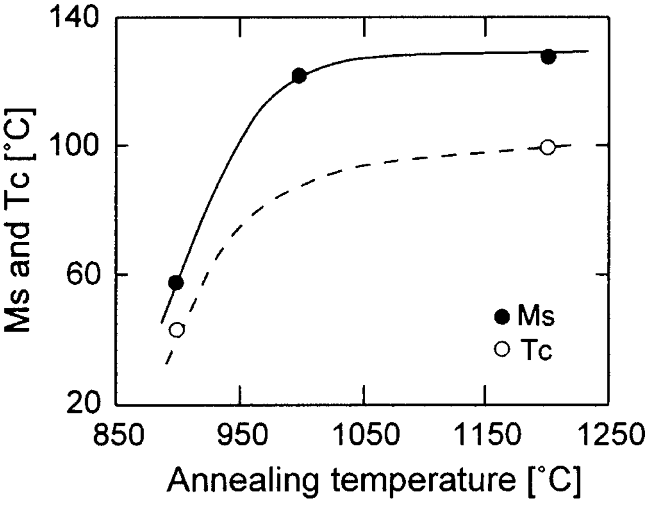

Fig. 6 Annealing temperature dependence of T_C and M_s of Co-25at.%Ni-30at.%Ga alloy

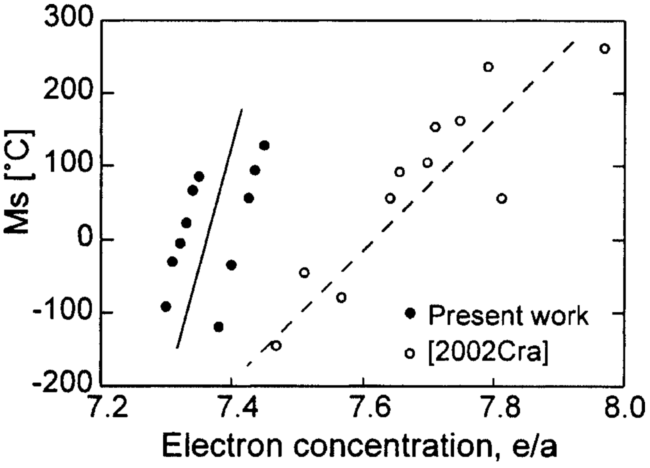

Fig. 7 Martensitic transition starting temperature M_s to valence electron concentration *e/a*

Co-Ni-Al [2001Oik1], Ni-Fe-Ga [2002Oik2], and Ni-Fe-Al [2003Oik1] bcc-ordered ferromagnetic shape memory alloys. The phase diagram is divided into three regions, as shown in Fig. 4. Alloys in region I martensitically transform from the ferromagnetic austenite to the ferromagnetic martensite. Alloys in region II transform from the paramagnetic austenite to the ferromagnetic martensite, namely, the first order-type magnetic transition from the paramagnetic to ferromagnetic is accompanied by the martensitic transition. In region III, the martensitic transformation occurs in the paramagnetic state, and the magnetic transition continues in further cooling.

From the experimental data, iso-T_C and iso-M_s temperatures for β single-phase alloy are evaluated, as shown in Fig. 5. The value of T_C increases with a decrease in both Ni and Ga contents, while M_s decreases with decreasing Ni content and with increasing Ga content. The hatched area in Fig. 5 shows the α + β two-phase region in the isothermal section at 1100 °C. The composition range of β phase exhibiting the martensitic transition from the ferromagnetic austenite state is located near the α + β two-phase region. Therefore, a small amount of the α-phase can be introduced in the β matrix by controlling the chemical composition and annealing temperature, which would bring about an improvement of ductility of the Co-Ni-Ga β based alloy as reported in the previous paper [2001Oik2].

The T_C and M_s of the α + β two-phase alloys are expected to depend on the annealing temperature due to the variation of the equilibrium composition of the β phase. Both the Ni and Ga compositions of β equilibrated with the α-phase decrease with increasing annealing temperature. The T_C and M_s of Co-25 at.% Ni-30 at.% Ga alloy with α + β two-phase structure are shown in Fig. 6.

Craciunescu et al. [2002Cra] recently showed good correlation between the M_s and the valence electron concentration *e/a*, which shows the same trend as in Ni-Mn-Ga alloys. The *e/a* dependence of M_s obtained from the β single-phase alloys in this work is plotted in Fig. 7 together with their data [2002Cra]. Here, the number of 3*d* + 4*s* electrons for Co and Ni atoms and that of 4*s* + 4*p* electrons for Ga atom is counted as the *e/a,* following the manner of the previous papers [2001Wut] [2002Cra]; the numbers of electron per atoms for Co, Ni, and Ga were assumed to be

Table 2 List of M_s, M_f, A_s, A_f, T_C, and *e/a* for Co-Ni-Ga β single-phase alloys

Alloy	M_s, °C	M_f, °C	A_s, °C	A_f, °C	T_C, °C	*e/a*
Co-50Ni-30Ga	…	…	…	…	−137	7.7
Co-40Ni-30Ga	…	…	…	…	−27	7.6
Co-32.5Ni-30Ga	…	…	…	…	48	7.525
Co-30Ni-30Ga	…	…	…	…	62	7.5
Co-25Ni-30Ga	128	95	140	183	99	7.45
Co-23.5Ni-30Ga	95	59	89	127	(a)	7.435
Co-22.5Ni-30Ga	56	28	56	84	117	7.425
Co-22.5Ni-30Ga(b)	−34	−67	−44	−3	…	…
Co-20Ni-30Ga	−35	−56	−26	−1	150	7.4
Co-18Ni-30Ga	−120	−138	−120	−103	181	7.38
Co-33Ni-32Ga	…	…	…	…	−56	7.41
Co-27Ni-32Ga	85	66	84	112	−8	7.35
Co-26Ni-32Ga	68	35	64	101	…	7.34
Co-25Ni-32Ga	22	−6	3	30	…	7.33
Co-24Ni-32Ga	−6	−40	−3	18	(a)	7.32
Co-23Ni-32Ga	−31	−47	−29	−8	−2	7.31
Co-22Ni-32Ga	−93	−104	−73	−65	…	7.3
Co-20Ni-32Ga	…	…	…	…	17	7.28
Co-18Ni-32Ga	…	…	…	…	67	7.26

(a) Magnetic transition temperature coincides with martensitic transition. (b) The alloy was aged at 300 °C for 1 h after quenched from 1200 °C.

9, 10, and 3, respectively. The M_s increases with increasing *e/a*, although the M_s curves of 32 at.% Ga and 30 at.% Ga alloy series do not fall on the same line. Therefore, it is rather difficult to explain the variation of the martensitic transition temperature of this system by the *e/a* ratio. The trend of M_s as a function *e/a* obtained in the present work is different from that of the data by Craciunescu et al. [2002Cra], as shown in Fig. 7. According to the present phase diagram of the Co-Ni-Ga system shown in Fig. 2, the alloys prepared by Craciunescu et al. [2002Cra] may be the α + β two-phase alloys; a chemical composition of β phase is different from an average composition of alloys. This is probably why our data of β single-phase alloys are inconsistent with the conclusion of Craciunescu et al. [2002Cra]. The martensitic and magnetic transition temperatures of all β single-phase alloys obtained in the present work are summarized in Table 2.

3.3 Crystal Structure of β Phase

Electron diffraction patterns in the β austenite phase of the Co-20at.%Ni-30at.%Ga alloy annealed at 1200, 600, and 300 °C are shown in Fig. 8. There is no significant difference between electron diffraction pattern of samples annealed at 1200 and 600 °C as shown in Fig. 8(a) and 8(b), where $\{100\}_{B2}$ ordered spots characterized as the B2 structure are observed in both the samples. However, the electron diffraction pattern of the sample aged at 300 °C shows the diffuse intensity characteristic of the ω phase precipitation found in other bcc-alloy systems as shown in Fig. 8(c). The dark-field image taken from the ω phase reflection shows the small particles 10-30 nm in diameter, as given in Fig. 8(d). The martensitic transition temperature is lowered after aging at 300 °C, as listed in Table 2. The characteristic reflections of Co_2NiGa $L2_1$ Heusler structure have not been observed in the current study. This result is consistent with the results of the XRD [1985Che] [1987Mik] and neutron analyses [1978Boo]. Therefore, it can be concluded that the crystal structure of the austenite phase is B2 structure.

Figure 9 shows the XRD patterns of the martensite phase obtained from the Co-30at.%Ni-30at.%Ga alloy powder rapidly cooled from 1200 °C. The spectrum peaks of the martensite phase are identified as $L1_0$ tetragonal structure with the small amount of the coexisting α-phase. The α-phase probably precipitates during the cooling process because the cooling rate from the solution treatment temperature of the powder without breaking the quartz tube is slower than that of the quenched bulk sample. The lattice parameters of the martensite phase have been determined to be $a_{L10} = 0.391$ nm, $c_{L10} = 0.315$ nm, and $c_{L10}/a_{L10} = 0.806$. These values are in accord with the data of Chernenko et al. [2004Che] estimated from the electron diffraction pattern and very close to that of Co-Ni-Al shape memory alloys [1996Kai] [2003Oik2].

3.4 Magnetic Properties of β Phase

The magnetization-external magnetic field curves of the martensite at −190 °C and the austenite phase at −50 °C for the Co-18at.%Ni-30at.%Ga β single-phase alloy are shown in Fig. 10(a). The magnetization curve of the austenite phase is more easily saturated than that of the martensite phase. This trend is similar to that of other ferromagnetic shape memory alloys; the martensite phase has higher magnetocrystalline anisotropy energy than the austenite phase. Figure 10(b) shows the composition dependence of the saturated magnetization I_s of the β single-phase alloys at

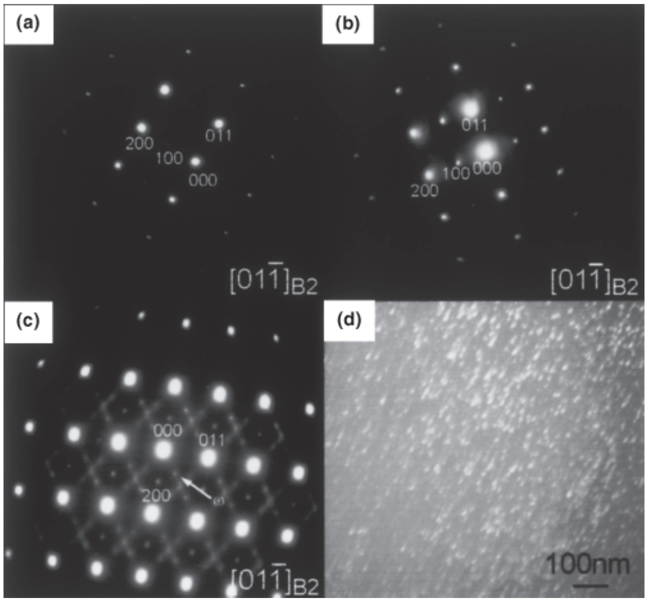

Fig. 8 Electron diffraction patterns in β phase of the Co-20at.%Ni-30at.%Ga alloy annealed at (a) 1200 °C for 4 h, (b) 600 °C for 30 days, and (c) 300 °C for 1 day; (d) TEM dark field image taken from ω phase reflection indicated by a white arrow in (c)

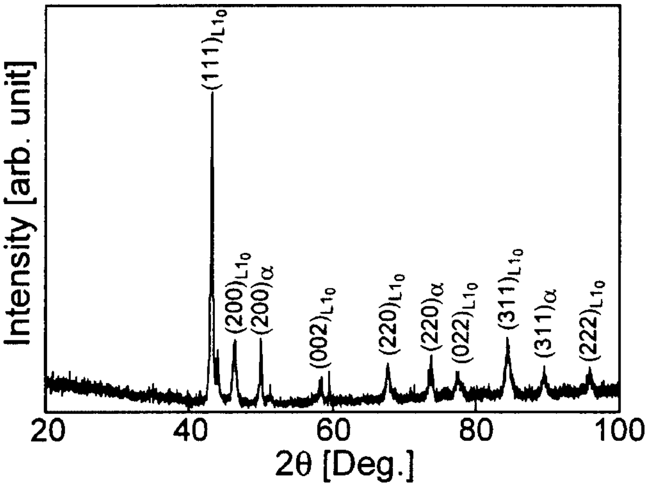

Fig. 9 XRD pattern obtained from Co-30at.%Ni-30at.%Ga powder cooled from 1200 °C

−190 °C in the 30 at.% Ga section. The I_s decreases monotonously with increasing Ni content.

It is known that the magnetization of transition metal alloy is closely related to an electron concentration such as the Slater-Pauling curve. The I_s values are plotted as a function of the electron concentration *e/a* together with the previous data of the Co-Ga [1970Boo] [1977Cyw] and Co-Ni-Ga [1978Boo] systems in Fig. 11. The I_s does not show a good correlation with *e/a*. Next, the generalized Slater-Pauling curve [1983Wil] is applied to the saturation magnetization and Curie temperature, as shown in Fig. 12. The values of magnetic valence Z_m are 1, 0, and −3 for Co, Ni, and Ga, respectively.[1983Wil] The I_s and T_C increase with an increase of Z_m and show good correlation with Z_m, as shown in Fig. 12(a) and (b), respectively. It should be noted that the T_C curves of the B2 and $L1_0$ do not fall on the same

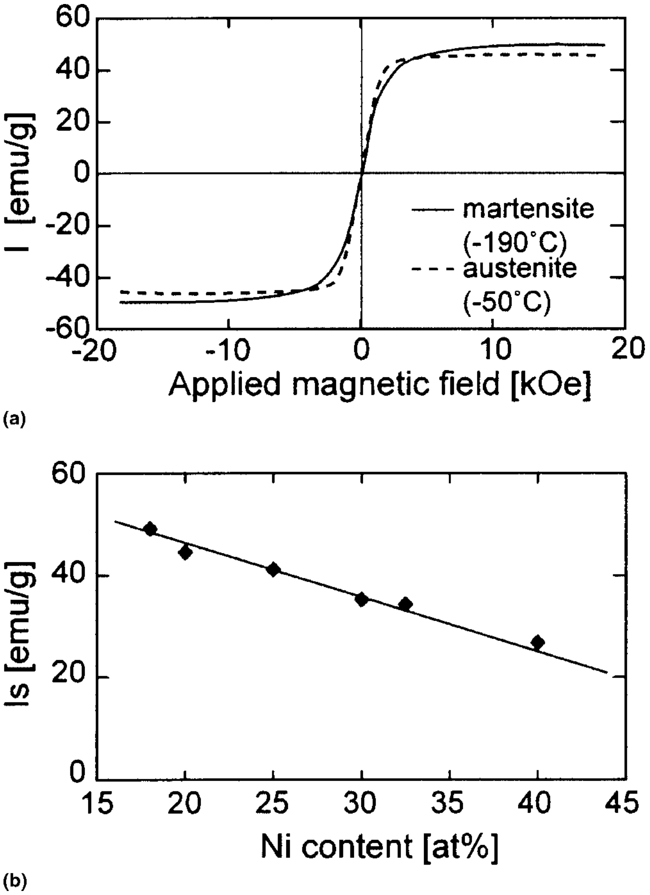

Fig. 10 (a) Magnetization *I* curves of martensite and austenite phase of Co-18at.%Ni-30at.%Ga β single-phase alloy; (b) composition dependence of saturation magnetization I_s at −190 °C in the Co-Ni-30 at.% Ga β single-phase alloys

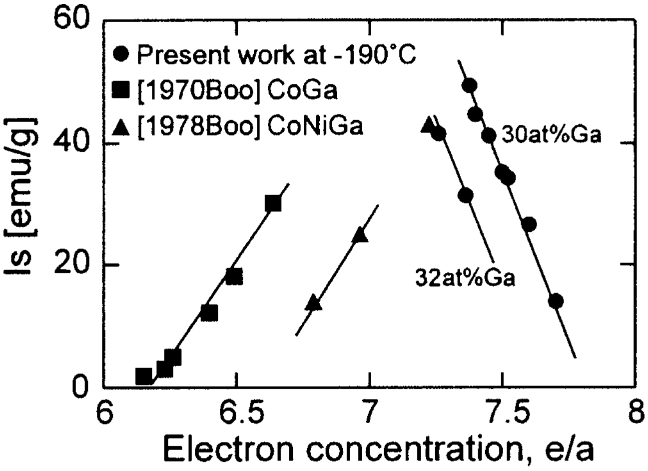

Fig. 11 Saturation magnetization I_s versus the average valence electron concentration *e/a*

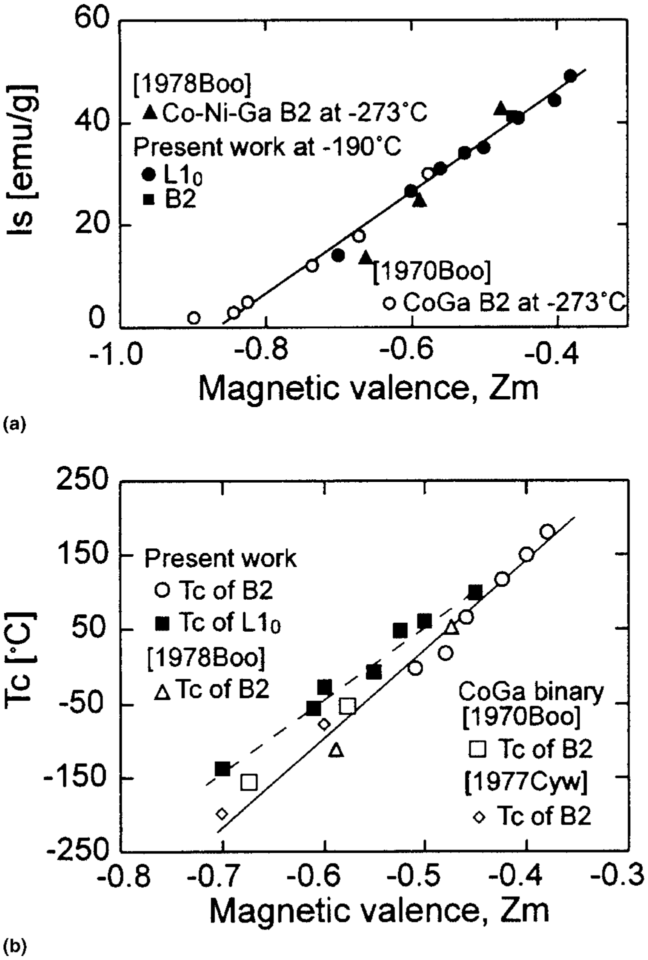

Fig. 12 Magnetic valence number Z_m dependence of (a) the saturation magnetization I_s and (b) the Curie temperature T_C

line with each other, and the T_C of $L1_0$ is higher than that of B2 [2001Oik1, 2001Oik2]. The Z_m is very useful for estimating the I_s and T_C of this system. The I_s data of β single-phase alloys obtained in the present work is summarized in Table 3.

4. Conclusions

The α(α′) + β phase equilibria, the martensitic and ferro/paramagnetic phase transformations in the β phase region of the Co-Ni-Ga system have been systematically investigated, and the following conclusions were made.

The α + β two-phase region of the Co-Ni-Ga system exists from the Co-Ga side to near the Ni-Ga side. The composition area of the β phase exhibiting the martensitic transition from the ferromagnetic austenite is located near the α + β two-phase boundary. Therefore, a small amount of the α-phase can be introduced in the β matrix by controlling the chemical composition and annealing temperature, which would bring about an improvement of the ductility of polycrystalline β alloys.

The Curie temperature T_C and the martensitic transition temperatures of the β single-phase alloys are sensitive to the Ni and Ga compositions. The value of T_C increases with decreases in both Ni and/or Ga contents, while the martensitic transition starting temperature M_s decreases with decreases in Ni content but with increases in Ga content. The T_C and M_s of the α + β two-phase alloy depend on the annealing temperature affecting variations in the chemical composition of β phase, increasing with an increase of annealing temperature.

Table 3 Saturated magnetization is at −190 °C and magnetic valence number Z_m for Co-Ni-Ga β single-phase alloys

Alloy	I_s, emu/g	Z_m
Co-50Ni-30Ga	14.15	−0.7
Co-40Ni-30Ga	26.83	−0.6
Co-32.5Ni-30Ga	34.31	−0.525
Co-30Ni-30Ga	35.25	−0.5
Co-25Ni-30Ga	41.14	−0.45
Co-20Ni-30Ga	44.52	−0.4
Co-18Ni-30Ga	49.19	−0.38
Co-28Ni-32Ga	31.29	−0.56
Co-18Ni-32Ga	41.44	−0.46

The crystal structures of the martensite and austenite phases are identified as $L1_0$ and B2 structures, respectively. Reflections expected from an $L2_1$ structure are not confirmed. Aging at 300 °C causes the precipitation of the nanoscale ω phase in the β matrix.

The saturated magnetization and T_C show good correlation with the average magnetic valence number of Co, Ni, and Ga atoms.

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