Invar Effect on Martensitic Transformations in Fe-Ni-C Alloys under Hydrostatic Pressure*

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Magnetic properties of Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C (at%) alloys have been examined by measuring magnetizations and forced volume magnetostrictions. Then, the M_s temperatures of those alloys under hydrostatic pressure have been determined by electrical resistivity vs temperature measurements up to 1.5 GPa. The results obtained were as follows. The M_s temperatures decreased with increasing pressure, but its decreasing manner was different between invar (Fe-28.4Ni-0.6C and Fe-22.6Ni-1.4C) and non-invar (Fe-18.6Ni-3.1C) alloys. The decrease in the non-invar alloy was linear with the increase of pressure, and the linear relation was consistent with that calculated by inserting the measured volume change associated with the transformation into a modified Patel-Cohen equation. On the other hand, the decrease in the invar alloys was not linear but accelerative with the increase of pressure, being largely different from a linear relation calculated from the modified Patel-Cohen equation. The non-linearity between the shift of M_s temperature and pressure in invar alloys was then concluded to be due to the invar nature of the ferromagnetic alloys. Magnetization measurements revealed that the amount of martensites formed under hydrostatic pressure decreased with increasing pressure. Optical microscopy showed the martensite morphology to be the same as that of thermally-induced transformations.

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I. Introduction

The influence of hydrostatic pressure on martensitic transformations has so far been examined by several workers⁽¹⁾⁻⁽³⁾. According to the previous examinations, the M_s temperature decreases in Fe-Ni⁽¹⁾ and Fe-Ni-C⁽²⁾ alloys whose volume changes associated with mar-

tensitic transformations are positive, while it increases in a Au-Cd alloy⁽³⁾ whose volume change is negative.

Recently, the present authors have examined in more detail the influence of hydrostatic pressure on martensitic transformations in Fe-Ni⁽⁴⁾, Fe-Ni-C⁽⁴⁾ and Cu-Al-Ni⁽⁵⁾ alloys, applying a higher pressure than in those previous examinations. As a result, it was confirmed that M_s temperatures of the former two ferrous alloys with positive volume changes decreased as previously reported, and that the latter Cu-Al-Ni alloy with a negative volume change exhibited an increase in M_s temperature, similarly to the Au-Cd alloy. However, the decreasing manner in M_s temperature of ferrous alloys was quite different from that in the previous examinations. That is, the measured hydrostatic pressure dependence of M_s temperature was largely different from the calculated one from a modified Patel-Cohen equation, especially on the higher pressure side. On the other hand, such a difference between measure-

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ment and calculation was not recognized in the Cu–Al–Ni alloy under the pressure range examined. The former ferrous alloys are ferromagnetic, while the latter Cu–Al–Ni alloy is diamagnetic. Thus, as to the hydrostatic pressure effect on martensitic transformations in ferrous alloys, it was concluded⁽⁵⁾ that other factors should be taken into consideration besides the volume change associated with the transformations, and that the other factors should be related to some magnetic properties of ferrous alloys. Of the magnetic properties, the invar nature is supposed to be the most effective, because it may be directly related to the volume change during martensitic transformations.

The present study has thus been conducted to confirm the above supposition by examining martensitic transformations in invar and non-invar Fe–Ni–C alloys under hydrostatic pressure.

II. Experimental Procedure

Fe–28.4Ni–0.6C, Fe–22.6Ni–1.4C and Fe–18.6Ni–3.1C (at%) alloys were prepared by melting in a high frequency induction furnace under argon atmosphere and by casting into a water-cooled iron mold. Each ingot of the alloys was homogenized at 1473 K for 86.4 ks (24 h), and then hot- and cold-rolled. From the rolled sheets, $3 \times 3 \times 0.3$, $2.5 \times 5 \times 0.3$ and $15 \times 18 \times 1.5$ (mm³) size specimens were cut for forced volume magnetostriction and electrical resistivity measurements and for X-ray diffraction, respectively. Those specimens were finally solution-treated at 1473 K for 10.8 ks in a silica capsule filled with argon, and then quenched into iced water.

The forced volume magnetostriction and magnetization measurements were carried out in order to confirm the magnetic properties of the alloys, especially the invar effect, the former measurement being done by the Fabri-Pérot interferometry method. Details of the interferometry were already described elsewhere⁽⁶⁾. The electrical resistivity measurement was carried out in order to determine M_s temperature of the alloys under hydrostatic pressure from 0 to 1.5 GPa by the four-probe

method under the pressure. The hydrostatic pressure was generated by a piston cylinder type of the pressure instrument. Details of the pressure generator was also described elsewhere⁽⁷⁾. The above three measurements were carried out at Research Center for Extreme Materials, Osaka University.

The amount of martensites formed at M_s temperature under hydrostatic pressure was determined by magnetization measurements, in the same way as before⁽⁴⁾. Morphology of the martensites was observed by optical microscopy. The etchant used for the observation was a 35% sodium pyrosulfite solution. Lattice parameters of the austenite and martensite phases were measured by an X-ray diffraction experiment in order to obtain the volume change associated with martensitic transformations, and the decrease in M_s temperature was calculated from the modified Patel-Cohen equation by using the volume change.

III. Results

1. Magnetic properties of three Fe–Ni–C alloys

M_s temperatures of the three Fe–Ni–C alloys under no pressure were first determined by electrical resistivity vs temperature measurements, and the magnetization of those alloys in their austenitic state at 293 K was measured in the same way as before⁽⁸⁾. The obtained M_s temperatures and spontaneous magnetization are shown in Table 1. Those M_s temperatures are consistent with those previously reported. The values of spontaneous magnetization indicate that Fe–28.4 Ni–0.6C and Fe–22.6Ni–1.4C alloys are ferromagnetic, although the mag-

Table 1 M_s temperatures and magnetization of austenite (at 293 K) of Fe–28.4Ni–0.6C, Fe–22.6Ni–1.4C and Fe–18.6Ni–3.1C alloys.

Composi- tion (at %)	Fe–28.4Ni– 0.6C	Fe–22.6Ni– 1.4C	Fe–18.6Ni– 3.1C
M_s (K)	213	246	243
M^y (293 K) (μ_B /atom)	0.65	0.05	0

netization of the latter alloy is very small, and that Fe-18.6Ni-3.1C alloy is paramagnetic.

According to the previous studies⁽⁹⁾⁻⁽¹¹⁾, the forced volume magnetostriction, $(\partial\omega/\partial H)$, of many polycrystal alloys with a cubic crystal structure, such as Fe-Ni alloys, is isotropic, and therefore it may be regarded as $3 \times (\partial(\Delta l/l)/\partial H)$, where $\Delta l/l$ is the elongation ratio of a specimen under a magnetic field H . It may not be unreasonable to assume that $(\partial\omega/\partial H)$ for the present Fe-Ni-C alloys is also isotropic, because the tetragonality of the alloys is not so large. Then, Δl has been measured by means of the Fabry-Pérot interferometry method and $(\partial\omega/\partial H)$ has been derived.

The $\Delta l/l$ measured as a function of the strength of magnetic field for the three Fe-Ni-C alloys at 293 K is shown in Fig. 1, and the $(\partial\omega/\partial H)$ obtained from $3 \times (\partial(\Delta l/l)/\partial H)$ is shown in Fig. 2 as a function of temperature. It is known from Fig. 2 that $(\partial\omega/\partial H)$ is larger for the ferromagnetic Fe-28.4Ni-0.6C and Fe-22.6Ni-1.4C alloys than that for the paramagnetic Fe-18.6Ni-3.1C alloy, whose value is the same as that in non-invar alloys⁽¹¹⁾, and that with increasing temperature, it linearly decreases in the Fe-28.4Ni-0.6C alloy, increases in the Fe-22.6Ni-1.4C alloy and is almost constant in the Fe-18.6Ni-3.1C alloy. The reason for the different behavior against temperature is not clear at present, but the large and small $(\partial\omega/\partial H)$ for the ferromagnetic and paramagnetic alloys, respectively, clearly indicate that the ferromagnetic

Fig. 1 Elongation ratio, $\Delta l/l$, plotted as a function of the strength of magnetic field for Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C (at%) alloys at 293 K; measured by the Fabry-Pérot interferometry method.

Fig. 2 Forced volume magnetostriction, $\partial\omega/\partial H$, plotted as a function of temperature for Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C alloys.

alloys are invar and the paramagnetic alloy is non-invar.

2. Pressure dependence of M_s temperature

Figure 3 shows the electrical resistivity vs temperature relations of two invar and one non-invar Fe-Ni-C alloys under the pressure of 1.0 GPa. There are seen sudden decreases in electrical resistivity at 169, 183 and 173 K for the Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C alloys, respectively. The decreased temperatures correspond to M_s of these alloys under the pressure of 1.0 GPa, and they are largely decreased from those, 213, 246 and 243 K, respectively, of those alloys under no pressure. Such decreased M_s temperatures have been measured under the pressures of 0.5, 1.0, 1.25 and 1.5 GPa, and they are shown in Table 2, together with the amount of the decrease, $\Delta M_s = M_s - M'_s$, where M'_s represents the transformation temperature under pressure. Figure 4 is the plot of ΔM_s as a function of pressure. It is known from the figure that ΔM_s increases with pressure for all the alloys. However, the increasing manner is different from alloy to alloy, that is, ΔM_s increases almost linearly for the non-invar alloy and cur-

Fig. 3 Electrical resistivity vs temperature relations measured for Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C alloys under the pressure of 1.0 GPa.

Table 2 M_s temperatures under no pressure and various pressures and differences between them of Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C alloys.

GPa	Fe-28.4Ni-0.6C		Fe-22.6Ni-1.4C		Fe-18.6Ni-3.1C	
	M_s (K)	ΔM_s (K)	M_s (K)	ΔM_s (K)	M_s (K)	ΔM_s (K)
0	213	0	246	0	243	0
0.5	191	22	216	30	208	35
1.0	169	44	183	63	173	70
1.25	143	70	163	83	153	90
1.5	111	102	141	105	133	110

vilinearly for the invar alloys with increasing pressure. It should also be noted in the figure that ΔM_s for the non-invar alloy is larger than that for the invar alloys over the pressure range examined. These will be discussed later.

3. Lattice parameters of austenite and martensite phases

In order to calculate the hydrostatic pressure dependence of ΔM_s following the modified Patel-Cohen equation, the volume changes as-

Fig. 4 Shift of transformation temperature, ΔM_s ($=M_s - M'_s$), plotted as a function of hydrostatic pressure for Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C alloys.

sociated with martensitic transformations in the three Fe-Ni-C alloys must be known, and they may be obtained from lattice parameters of the austenite and martensite phases. The lattice parameters have then been measured by X-ray diffraction, and are shown in Fig. 5 as a function of temperature, (a), (b) and (c) being for a parameter of the austenite phase and a and c parameters of the martensite phase, respectively. In (c) the c parameter for the Fe-28.4Ni-0.6C alloy is not shown, because of a small tetragonality due to the low carbon content. All the lattice parameters measured increase with increasing temperature, although the increasing ratio is a little different from alloy to alloy.

From the measured lattice parameters, the volumes of the austenite and martensite phases under no pressure, $V^\gamma(T, 0)$ and $V^{\alpha'}(T, 0)$, respectively, were obtained as a function of temperature:

$$V^\gamma(T, 0) = 1/4 \cdot [3.604 \{1 + 2.59 \times 10^{-5} \times (T - 301)\}^3 \times 10^{-30} \text{ m}^3,$$

$$V^{\alpha'}(T, 0) = 1/2 \cdot [(2.862)^2 \cdot (2.956) \times \{1 + 1.00 \times 10^{-5}(T - 301)\}^2 \times \{1 + 3.65 \times 10^{-5}(T - 301)\} \times 10^{-30} \text{ m}^3,$$

for Fe-18.6Ni-3.1C alloy,

$$V^\gamma(T, 0) = 1/4 \cdot [3.594 \{1 + 1.96 \times 10^{-5}$$

Fig. 5 Temperature dependence of lattice parameters of the austenite and martensite phases in Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C alloys.

$$\times (T-301)]^3 \times 10^{-30} \text{ m}^3,$$

$$V^{\alpha'}(T, 0) = 1/2 \cdot [(2.865)^2 \cdot (2.911) \\ \times \{1 + 1.36 \times 10^{-5}(T-301)\}^2 \\ \times \{1 + 2.96 \times 10^{-5}(T-301)\}] \\ \times 10^{-30} \text{ m}^3,$$

for Fe-22.6Ni-1.4C alloy,

$$V^{\gamma}(T, 0) = 1/4 \cdot [3.586\{1 + 9.31 \times 10^{-6} \\ \times (T-303)\}]^3 \times 10^{-30} \text{ m}^3,$$

$$V^{\alpha'}(T, 0) = 1/2 \cdot [2.870\{1 + 1.66 \times 10^{-5} \\ \times (T-303)\}]^3 \times 10^{-30} \text{ m}^3,$$

for Fe-28.4Ni-0.6C alloy.

4. Amount and morphology of martensites formed under pressure

The amount of martensites induced at the

decreased M_s temperatures under various hydrostatic pressures has been determined from magnetization measurements in the same way as in the previous study⁽⁴⁾. The martensite amounts thus determined for the three alloys at the respective decreased M_s temperatures are plotted in Fig. 6 as a function of pressure. It is to be noted in the figure that the amount of martensites formed under pressure decreases with increasing pressure for all the alloys. These results are consistent with the previous study⁽⁴⁾, and may suggest that the nucleation and growth of martensites are suppressed as pressure increases and/or temperature decreases.

Figure 7 shows optical micrographs of thermally-induced lenticular martensites, (a), (c) and (e), formed in the three Fe-Ni-C alloys by cooling a little below their respective M_s temperatures and those of martensites formed under the pressure of 1.5 GPa, (b), (d) and (f). Formation temperatures and ΔT are also inscribed in each photograph. It should be noted from the figure that the morphology of mar-

Fig. 6 Calculated amount of martensites formed at M_s' under pressure, plotted as a function of pressure for Fe-28.4Ni-0.6C, Fe-22.6Ni-1.4C and Fe-18.6Ni-3.1C alloys.

Fig. 7 Optical micrographs of thermally-induced martensites, (a), (c) and (e), and hydrostatic pressure-induced ones, (b), (d) and (f), (a) and (b) being of Fe-28.4Ni-0.6C alloy, (c) and (d) of Fe-22.6Ni-1.4C alloy, and (e) and (f) of Fe-18.6Ni-3.1C alloy. Formation temperature, T , its difference from M_s , ΔT , and pressure, P , for martensites formed under hydrostatic pressure are shown for each photograph.

tensites formed under pressure is very similar to that of thermally-induced ones irrespective of the formation temperature or ΔT . This result is also consistent with the previous study⁽⁴⁾.

IV. Discussion

Patel and Cohen proposed an equation to estimate ΔM_s as a function of hydrostatic pressure. However, they did not take account of temperature and pressure dependences of the volume changes of austenite and martensite phases. Then, the present authors have proposed a more precise equation⁽⁴⁾⁽⁵⁾ instead of the Patel-Cohen equation. The precise equation is as follows:

$$\Delta G(M_s) - \Delta G(T) = \{V^\gamma(T, 0) - V^{\alpha'}(T, 0)\} \cdot P + 1/2 \cdot \{V^{\alpha'}(T, 0)/B^{\alpha'}(T) - V^\gamma(T, 0)/B^\gamma(T)\} \cdot P^2,$$

where $\Delta G(M_s)$ and $\Delta G(T)$ represent the difference in the Gibbs chemical free energy be-

tween the austenite and martensite phases under no pressure at M_s and at a lower temperature T , respectively, and $V^\gamma(T, 0)$ and $V^{\alpha'}(T, 0)$ the volumes of the austenite and martensite phases, respectively, at T and under no pressure. $B^\gamma(T)$ and $B^{\alpha'}(T)$ are the bulk moduli of the austenite and martensite phases at T , respectively. The temperature T satisfying the above equation is nothing but the decreased M_s temperature, M'_s , under the pressure P .

By using the above equation, the hydrostatic pressure dependence of ΔM_s was calculated in the same way as in the previous study⁽⁴⁾. That is, the Gibbs chemical free energy difference were obtained from the equations derived by Kaufman and Cohen⁽¹²⁾, Korenko⁽¹³⁾ and Fisher *et al.*⁽¹⁴⁾⁽¹⁵⁾ $V^\gamma(T, 0)$ and $V^{\alpha'}(T, 0)$ obtained in the preceding section were used as those of the above equation. The bulk modulus B^γ was obtained by referring to the values previously measured by Papadakis and Reed⁽¹⁶⁾, and $B^{\alpha'}$ was substituted with that for pure iron, although this is a rough approximation.

The hydrostatic pressure dependence of ΔM_s

Fig. 8 Calculated pressure dependence of ΔM_s together with the measured one of Fe-28.4Ni-0.6C (---), Fe-22.6Ni-1.4C (---) and Fe-18.6Ni-3.1C (---) alloys.

thus calculated for one non-invar and two invar Fe-Ni-C alloys are shown with the dot-dot-dashed, dot-dashed and dashed lines, respectively, in Fig. 8, together with those experimentally measured. It is known from the figure that the calculated and measured dependences of ΔM_s show good agreement for the non-invar Fe-18.6Ni-3.1C alloy, but they are largely different for the invar Fe-22.6Ni-1.4C and Fe-28.4Ni-0.6C alloys, especially for the latter alloy under the pressure higher than 1.0 GPa. It may then be concluded that the large difference between the calculation and measurement is due to the invar nature of ferromagnetic alloys. Therefore, the influence of hydrostatic pressure on martensitic transformations in invar alloys should be said to consist of an invar effect and the volume change effect associated with the transformation.

By the way, it was noticed before that ΔM_s for the non-invar Fe-18.6Ni-3.1C alloy was larger than that for the invar Fe-22.6Ni-1.4C and Fe-28.4Ni-0.6C alloys. This fact and the above difference between calculation and measurement will be discussed on the basis of Fig. 9 which is a schematic representation of the temperature dependences of the volumes of the austenite and martensite phases in ferrous invar alloys. When a ferrous invar alloy, such as Fe-Ni or Fe-Ni-C, is subjected to a paramagnetic to ferromagnetic transition at the Curie temperature T_c , the alloy exhibits an

Fig. 9 Schematic volume change of austenite and martensite phases as a function of temperature.

invar nature, because an expansion due to the spontaneous volume magnetostriction cancels the thermal contraction. That is, the volume of the austenite phase changes along the solid line indicated with V_p^β and V_f^β above and below T_c , respectively. On the other hand, the volume of the martensite phase changes along the solid line indicated with $V^{\alpha'}$. If the alloy is subjected to a martensitic transformation at T lower than T_c , the volume change associated with the martensitic transformation is $\Delta V_f = V^{\alpha'} - V_f^\beta$. However, if the alloy were paramagnetic non-invar, the volume change associated with the transformation at T would be $\Delta V_p = V^{\alpha'} - V_p^\beta$. Since ΔV_p is larger than ΔV_f , ΔM_s which is proportional to a bilinear product of ΔV with pressure P may be larger in the paramagnetic non-invar alloy than in the ferromagnetic invar alloy, as actually observed in Figs. 4 and 8.

Next, discussion will be done on the fact that ΔM_s in invar alloys acceleratingly increases and largely deviates from the calculated linear relation. According to a previous study⁽¹⁷⁾, the spontaneous magnetostriction, $\omega_s = (V_f^\beta - V_p^\beta) / V_p^\beta$, is proportional to the square of spontaneous magnetization M which becomes smaller with increasing pressure. Therefore, the volume expansion due to the invar nature, $V_f^\beta - V_p^\beta (= \omega_s \cdot V_p^\beta)$ acceleratedly decreases with increasing pressure, and hence the volume change associated with martensitic transforma-

tion, $\Delta V = V^{\alpha'} - V_f^{\gamma}$ inversely increases, causing an accelerating increase in ΔM_s under the pressure, as also observed in Figs. 4 and 8. Thus, the accelerated increase of ΔM_s in invar alloys was explained by taking into consideration the pressure dependence of the spontaneous magnetization and/or volume magnetostriction. However, such a pressure (may be also temperature) dependence of ω_s and M should be quantitatively examined in order to more clearly explain the accelerated increase of ΔM_s in invar alloys, especially the extraordinary increase of ΔM_s in the Fe-28.4Ni-0.6C alloy under the pressure higher than 1.0 GPa.

The Curie temperature T_c may be expected to decrease with increasing pressure. If so, V_f^{γ} may be shifted downwards, and the volume change associated with martensitic transformation at T lower than T_c may be increased compared with that under no pressure. Then, the pressure dependence of such a decrease of T_c should also be examined more quantitatively. These examinations are now being carried out.

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