Effect of Magnetic Fields on Athermal and Isothermal Martensitic Transformations in Fe-Ni-Mn Alloys[†]

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The effect of magnetic fields on the athermal martensitic transformation in an Fe-31.4Ni-0.5Mn alloy (mass%), whose M_s temperature is about 195 K, and on the isothermal one in an Fe-24.9Ni-3.9Mn alloy (mass%), whose nose temperature is about 153 K, have been examined in order to clarify the difference between the athermal and isothermal kinetics of martensitic transformations by measuring magnetic field susceptibility and magnetization, and by observing optical microstructure, applying pulsed ultra high magnetic fields up to 31 MA/m. As a result, the following characteristics were found: The austenitic state in the former alloy was ferromagnetic, whereas that in the latter alloy was micromagnetic and/or spin glass. Even in the latter alloy, martensitic transformation was induced instantaneously under pulsed magnetic fields higher than a critical one over a wide temperature range, as in the former alloy. This result suggests that the originally isothermal kinetics of martensitic transformation is changed to the athermal one under high magnetic fields. Optical microscopy showed that respective isothermal martensite plates of the Fe-24.9Ni-3.9Mn alloy under no magnetic field grew gradually during isothermal holding. Moreover, morphology of the magnetic field-induced martensites of both the alloys was almost the same as that of thermally-induced ones, irrespective of the formation temperature. A thermodynamic calculation for the critical magnetic field vs. temperature relation was done for both the alloys by using an equation previously proposed, and the calculated relations were all in good agreement with the experimental ones in the wide temperature range examined.

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I. Introduction

It is well known that martensitic transformations may be classified into two groups, athermal and isothermal ones, with respect to their temperature and time dependences of the amount of martensites. That is, the amount of martensites is a function of only temperature in the former group, but of both temperature and time in the latter group. However, materials exhibiting the latter isothermal martensitic transformation are very few in number, and Fe-Ni-Mn alloys are known to be the typical of such materials.

There is a view⁽¹⁾⁽²⁾ that the isothermal transformation is rather general and the athermal one is its special case,

considering that the incubation time needed for the athermal transformation might be undetectably short. However, the view has not been verified yet, although the verification may give important information on the substantial problem of martensitic transformations, such as the nucleation and growth mechanism and the origin of transformations. Therefore, further investigations should be needed for the verification of the above view. As for such investigations, it may be very important to examine the effects of external forces other than temperature, whose effect has only been examined so far on athermal and isothermal martensitic transformations in Fe-Ni-Mn alloys. Because the martensitic transformations are known to be influenced also by the other external forces, such as uniaxial stress⁽³⁾⁽⁴⁾, hydrostatic pressure⁽³⁾⁽⁵⁾⁻⁽⁷⁾ and magnetic field⁽⁸⁾⁻⁽¹⁰⁾, in their transformation temperatures, crystal structure, amount and morphology. The examination of the effect of those external forces on martensitic transformations, therefore, may clarify finally the substantial problems of the martensitic transformations.

Many studies have thus been done on the effect of

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magnetic fields on martensitic transformations in several ferrous alloys and steels, especially by a Russian group⁽⁸⁾ and the authors' group⁽⁹⁾⁽¹⁰⁾. As a result, much useful information has been obtained about the effect of magnetic fields. Especially, we have clarified that magnetic effects on athermal martensitic transformations consist of cumulative three ones, magnetostatic, high field susceptibility and forced volume magnetostriction⁽⁹⁾, and we proposed the following equation to estimate the relation between the critical magnetic field for inducing martensites and the transformation start temperature:

$$\Delta G(M_s) - \Delta G(T) = -\Delta M(T) \cdot H - (1/2)\chi_h^p \cdot H^2 + \varepsilon_0 \times (\partial\omega/\partial H) \cdot H \cdot B, \quad (1)$$

where $\Delta G(M_s)$ and $\Delta G(T)$ represent the difference in Gibbs chemical free energy between the austenite and martensite phases at M_s and T temperatures, respectively, $\Delta M(T)$ the difference in spontaneous magnetization between the austenitic and martensitic states at T , H the magnetic field, χ_h^p the high magnetic field susceptibility in the austenite phase, ε_0 the transformation strain, ω the forced volume magnetostriction and B the austenitic bulk modulus. The first, second and third terms on the right-hand side of eq. (1) represent the energies due to the magnetostatic, high field susceptibility and forced volume magnetostriction effects, respectively. H and T satisfying the above equation are nothing but the critical magnetic field, H , at temperature, T . Moreover, the propriety of the equation has been confirmed for many alloy systems exhibiting the athermal martensitic transformation. However, it has not been confirmed yet for isothermal martensitic transformations although it should be needed, as mentioned. Only one study has been concerned with the isothermal martensitic transformation in an Fe-Ni-Mn alloy under a low static magnetic field⁽¹¹⁾, but no typical information was obtained as for the above problem. Furthermore, no study has been reported on the effect of magnetic fields on the athermal martensitic transformation in Fe-Ni-Mn alloys.

The purpose of the present study, therefore, is to examine in detail the effect of magnetic fields on the athermal and isothermal martensitic transformations in Fe-Ni-Mn alloys in the following way: The first is to examine athermal and isothermal characteristics in the Fe-Ni-Mn alloys, such as M_s and nose temperatures, transformation strain, spontaneous magnetization, magnetic field susceptibility, forced volume magnetostriction and morphology of martensites. The second is to obtain critical magnetic fields for inducing martensites at various temperatures in those alloys by magnetization measurements under pulsed magnetic fields up to 31 MA/m. The last is to calculate the critical magnetic field vs. temperature relations in those alloys by putting the measured physical quantities into eq. (1), and is to compare the calculated relations with the experimentally measured ones.

II. Experimental Procedure

The alloys examined were Fe-31.4Ni-0.5Mn and Fe-24.9Ni-3.9Mn (mass%) alloys, which exhibit athermal and isothermal martensitic transformations, respectively, and were produced by an argon arc-melting method. The alloys will be labeled alloys A and B, respectively, for simplicity. The ingots thus obtained were hot-forged, homogenized at 1373 K for 8.64×10^4 s and hot-rolled into sheets. From the sheets, specimens were cut into various sizes as follows depending on the kinds of measurements: 3 mm $\times$ 6 mm $\times$ 0.4 mm for electrical resistivity measurements of alloy A to obtain its M_s temperature, 3 mm $\times$ 6 mm $\times$ 0.4 mm for the isothermal holding measurements of alloy B to obtain its $T T T$ diagram, 14 mm $\times$ 18 mm $\times$ 1 mm for the X-ray diffraction measurements of both alloys to obtain their transformation strains, 3 mm $\times$ 12 mm $\times$ 0.4 mm for the spontaneous magnetization, 3 mm $\times$ 3 mm $\times$ 0.4 mm for the magnetic field susceptibility and 3 mm $\times$ 5 mm $\times$ 0.3 mm for the forced volume magnetostriction measurements for both alloys to know their magnetic properties and 3 mm $\times$ 12 mm $\times$ 0.4 mm for the magnetization measurements of both alloys under pulsed magnetic fields up to 31 MA/m. The specimens were finally solution-treated at 1273 K in silica capsules filled with argon, and then quenched into water. After the magnetization measurements, morphology of the martensites was observed by optical microscopy. The etchant used for the observation was a 35% sodium pyrosulfite solution. The magnetic field susceptibility, forced volume magnetostriction were measured by a Faraday type method and Fabry-Pérot interferometry method, respectively, and details of these measurements were already described elsewhere⁽⁹⁾. Magnetization measurements were carried out at Research Center for Extreme Materials, Osaka University. The details of the ultra high magnetic field instrument have been reported elsewhere⁽¹²⁾. Chemical analysis of the alloy compositions were carried out by using the rest part of the 0.4 mm thick sheets, which were subjected to the same heat-treatment as above in order to avoid a discrepancy in composition between the chemically analyzed and experimentally used specimens. The alloy compositions thus analyzed are the ones mentioned at the beginning of this section.

III. Results

1. $T T T$ diagram, transformation strain, spontaneous magnetization, magnetic field susceptibility and forced volume magnetostriction

Prior to measurements under pulsed high magnetic fields, some physical properties of alloys A and B have been examined, such as transformation temperature, $T T T$ diagram, transformation strain, spontaneous magnetization, magnetic field susceptibility and forced volume magnetostriction. First of all, the electrical

resistivities have been measured as a function of temperature for both alloys in the range between 293 and 77 K. As a result, an abrupt decrease in resistivity was recognized at 195 K for alloy A, which is similar to those in previous Fe-Ni alloys⁽¹³⁾ exhibiting an athermal martensitic transformation, while no change is recognized for alloy B. This suggests that the alloy A certainly exhibits an athermal transformation kinetics at 195 K, which corresponds to the M_s temperature of the alloy, but alloy B is not. In fact, optical microscopy shows that martensite plates in the alloy A were certainly induced at this temperature (whose morphology will be shown later.), but not for the B one at any temperature. Then, alloy B has been isothermally held at several temperatures between 4.2 and 293 K. A typical result of the measurements of martensite amount vs. temperature relation is shown in Fig. 1(a), where the amount of martensites was measured

by counting the martensite plates in optical microscopy. It should be noted in the figure that the amount of martensites depends not only on temperature but also on time, clearly indicating that the alloy B exhibits an isothermal kinetics of martensitic transformation. From (a), a $T-T-t$ diagram has been obtained, which represents 0.1% volume fraction of martensites, and is shown with the closed circles connected by the solid line in Fig. 1(b). It should be noted in (b) that the $T-T-t$ diagram clearly forms a C-curve whose nose temperature is about 153 K and the C-curve is asymmetric, that is, the time required for the formation of 0.1% volume fraction of martensites is different between the higher and the lower temperatures sides. This result is the same as that in a previous study⁽¹⁴⁾, and may give important information on isothermal kinetics of martensitic transformations, which will be discussed later.

Incidentally, respective martensite plates under no magnetic field were observed to grow gradually during isothermal holding by optical microscopy, as shown in Fig. 2(a) to (f), as has already been observed by an electron microscopy⁽¹⁵⁾. Transformation strains for alloy A and another Fe-22.7Ni-3.9Mn alloy (mass%), as a substitute for alloy B, have been calculated from their lattice parameters of the austenitic and martensitic phases measured by X-ray diffraction with a cold stage. The transformation strains will be used to calculate the relation between critical magnetic field and temperature, which will be described later. The reason for using the other Fe-22.7Ni-3.9Mn alloy as a substitute is that the alloy has nearly the same composition as alloy B and also exhibits an isothermal transformation. Moreover, the amount of martensites in the substitute alloy is enough to be detected by X-ray diffraction, although that in alloy B is not enough in spite of the long isothermal holding. However, the substitute alloy was not used throughout the present work, because the incubation time of the martensite formation in the substitute alloy is much shorter than that in the alloy B, and therefore the amount of martensites in the former alloy is largely increased during specimen setting at a temperature before applying the magnetic field. Such a large increase is inappropriate in examining only the influence of magnetic fields on the isothermal martensitic transformation. The transformation strains thus calculated are shown in Fig. 3 as a function of temperature for alloy A and the substitute of alloy B. As known from the figure, the transformation strain for the alloy A decreases a little, but that for the substitute of alloy B increases with decreasing temperature. The former decrease may be due to the invar effect taking account of the fact that coefficient of linear expansion of the austenitic phase in the alloy A is quite small (8.74×10^{-6}), as in a previous Fe-29.9 at%Ni invar alloy⁽⁷⁾⁽⁹⁾.

Magnetization in the austenitic state of the two alloys under low magnetic fields has been measured. As a result, the austenitic states of alloys A and B were ferromagnetic because of the existence of spontaneous magnetization, as shown in Fig. 4. However, the temperature depend-

Fig. 1 Amount of martensites as a function of holding time, (a), and $T-T-t$ diagram of the isothermal martensitic transformation, (b), in an Fe-24.9Ni-3.9Mn alloy, the closed circles connected with the solid line in (b) being the measured ones from (a).

Fig. 2 *In-situ* optical micrographs taken at room temperature after the isothermal holding at 153 K, showing the gradual growth of a martensite plate in an Fe-24.9Ni-3.9Mn alloy. Holding time, t , at 153 K is inscribed in each photograph.

ence of spontaneous magnetization in alloy B seems to be quite strange because spontaneous magnetization at 4.2 K is a little lower than that at 77 K, being different from those of normal ferromagnetic materials. Then, in order to know the magnetic state of alloy B in more detail, magnetic field susceptibility of the alloy has been measured by a Faraday type magnetic balance method, applying the low static magnetic field of 7.9×10^{-2} MA/m. A typical result of the measurements is shown in Fig. 5, as a function of temperature. It is noted that the magnetic field susceptibility has a broad peak around 77 K, which is reversible in the temperature range measured. This suggests that the austenitic state in the alloy exhibits a mictomagnetism and/or spin glass (ferromagnetic and antiferromagnetic state coexist), as previously reported

in another Fe-Ni-Mn alloy⁽¹⁶⁾. On the other hand, spontaneous magnetizations of the martensitic state in the alloys are obtained by referring to the Slater-Pauling curve⁽¹⁷⁾. The values for the alloys A and B are $1.9\mu_B$ and $2.1\mu_B$ at 0 K, respectively, and considered to be valid in the temperature range where the magnetization has been measured, because the Curie temperatures of those alloys are much higher than the measured temperatures. Temperature dependences of the forced volume magnetostriction in the austenitic state of the alloys have been obtained by Fabry-Pérot interferometry with the He-Ne laser of $0.6328 \mu\text{m}$ in wavelength, and are shown in Fig. 6 for both the alloys. As known from the figure, alloy A has a large forced volume magnetostriction, as in a previous Fe-29.9 at%Ni invar alloy⁽⁹⁾, while that of alloy

Fig. 3 Transformation strains associated with the martensitic transformations in Fe-31.4Ni-0.5Mn and Fe-22.7Ni-3.9Mn alloys (as substitute of Fe-24.9Ni-3.9Mn alloy), plotted as a function of temperature.

Fig. 4 Spontaneous magnetizations in the austenitic state, M_0^a , for Fe-31.4Ni-0.5Mn and Fe-24.9Ni-3.9Mn alloys, plotted as a function of temperature.

B is comparatively small and one half smaller than that of the former.

2. Critical magnetic fields for inducing martensites and amount and morphology of magnetic field-induced martensites

Magnetization $M(t)$ has been measured as a function of magnetic field $H(t)$ in one pulse. Typical $M(t)-H(t)$ curves are shown in Fig. 7(a) and (b) for alloy A at 218 K, which is 23 K higher than M_s temperature, and for alloy B at 4.2 K, respectively. In (a), an instantaneous increase in magnetization is recognized at a certain strength of magnetic field (indicating with an arrow), as in previous alloys⁽⁹⁾⁽¹³⁾ exhibiting an athermal martensitic

Fig. 5 Magnetic field susceptibility, χ , of an Fe-24.9Ni-3.9Mn alloy, plotted as a function of temperature.

Fig. 6 Forced volume magnetostrictions for Fe-31.4Ni-0.5Mn and Fe-24.9Ni-3.9Mn alloys, plotted as a function of temperature.

transformation. Such an increase in magnetization could not be observed if the maximum strength of an applied magnetic field was lower than the certain strength of magnetic field. Therefore, the certain strength of magnetic field corresponds to the critical one for inducing the martensitic transformation. A similar increase in magnetization (indicating with an arrow) is recognized in alloy B, as seen in (b), although under no magnetic field no isothermal martensitic transformation was observed in the alloy at 4.2 K, despite isothermal holding for the longer periods more than 8.64×10^4 s. Similar $M-H$ curves were obtained at temperatures higher than 4.2 K. These results suggest that the isothermal kinetics of martensitic transformation is changed to an athermal one under high magnetic fields. This fact may give important information on the kinetics of martensitic transformation. Figure 8 shows the relations between the critical magnetic fields and transformation temperatures (indicated with the solid and open circles) for both alloys A and B. As known from the figure, the relation for alloy B

Fig. 7 Magnetization vs. magnetic field curves of an Fe-31.4Ni-0.5Mn alloy at 218 K, (a), and an Fe-24.9Ni-3.9Mn alloy at 4.2 K, (b), the magnetization being increased instantaneously at a critical magnetic field indicated with an arrow.

Fig. 8 Critical magnetic field vs. temperature relations of Fe-31.4Ni-0.5Mn and Fe-24.9Ni-3.9Mn alloys, and the dotted lines represent the calculated ones.

does not exhibit any nose temperature, being different from the isothermal characteristic under no magnetic field, and decreases with decreasing temperature. Similar decrease is observed in alloy A and previous alloys exhibiting an athermal martensitic transformation⁽⁹⁾⁽¹³⁾, but the decrease is linear toward M_s .

Fig. 9 High-field susceptibilities, χ_h^p , plotted as a function of temperature.

Fig. 10 Amounts of magnetic field-induced martensites in Fe-31.4Ni-0.5Mn and Fe-24.9Ni-3.9Mn alloys under the magnetic field of 29.4 MA/m, plotted as a function of temperature.

Magnetic high field susceptibility in the austenitic phase, χ_h^p , was obtained from the $M(t) - H(t)$ curves shown in Fig. 7(a) and (b), and their temperature dependences are shown in Fig. 9 for alloys A and B. The amount of magnetic field-induced martensites formed under the magnetic field of 29.4 MA/m has been calculated in the same manner as in the previous study⁽¹³⁾. The calculated martensite amounts are shown in Fig. 10. It is noted from the figure that the martensite amount for both alloys increases with decreasing temperature, and that for alloy B is about 65% at 4.2 K, although any martensites could not be observed during long isothermal holding at 4.2 K under no magnetic field.

Figures 11 and 12 show optical micrographs of thermal-

Fig. 11 Optical micrographs of thermally-induced martensites, (a), and magnetic field-induced martensites, (b), in an Fe-31.4Ni-0.5Mn alloy.

Fig. 12 Optical micrographs of magnetic field-induced martensites, (a) and (b), and thermally-induced martensites, (c) and (d), in an Fe-24.9Ni-3.9Mn alloy.

ly- and magnetic field-induced martensites in alloys A and B, respectively, (a) in Fig. 11 and (c) and (d) in Fig. 12 being for thermally-induced martensites, and formation temperature and magnetic field being inscribed on each photograph. It should be noted from the figures that morphology of the magnetic field-induced martensites in both alloys are similar to that of thermally-

induced martensites, irrespective of the formation temperature.

IV. Discussion

Based on eq. (1), relation between critical magnetic field and transformation start temperature has been

calculated for the two alloys used, and the calculated relation was compared with the experimental one. First of all, we have calculated as to alloy A exhibiting an athermal martensitic transformation. In the calculation, the physical quantities involved in eq. (1), $\Delta M(T)$, χ_h^p , ε_0 and ω , were set to be the ones measured in the present study, although the bulk modulus of the austenitic phase, B , and the difference in Gibbs chemical free energy, $\Delta G(T)$, were referred to a previous study⁽¹⁸⁾ and followed the equation already derived by Kaufman⁽¹⁹⁾, respectively. The result thus calculated is shown with the dashed line in Fig. 8, being almost in good agreement with the measured one, as in previous studies⁽⁹⁾⁽¹³⁾. The same calculation has been made for alloy B exhibiting an isothermal martensitic transformation, putting the physical quantities obtained in the present work and previously obtained B and $\Delta G(T)$ into eq. (1). In the calculation, however, the driving force for making the transformation start, which corresponds to $\Delta G(M_s)$ in an athermal one, can not be calculated even when $\Delta G(T)$ is given in this case, because no M_s temperature exists. Therefore, the driving force for making the transformation start has been obtained in the following way. That is, considering that eq. (1) holds for an isothermal martensitic transformation, the driving force for making the martensitic transformation start, $\delta (= \Delta G(M_s)$ for an athermal martensitic transformation), may be written as

$$\delta = \Delta G(T) - \Delta M(T) \cdot H - (1/2) \chi_h^p \cdot H^2 + \varepsilon_0 \cdot (\partial \omega / \partial H) \cdot H \cdot B. \quad (2)$$

Thus, δ can be estimated by putting the measured critical magnetic field and other physical quantities into eq. (2), and the value thus obtained is 1574 J/mol under the critical magnetic field of 29.4 MA/m at 223 K, being much higher than $\Delta G(0)$ (1384 J/mol). This value is fixed during the calculation. The calculated critical magnetic field vs. temperature relation is shown with a dotted line in Fig. 8, also being almost in good agreement with the experimental one in the wide temperature range between 4.2 K and 223 K. This means that the effect of magnetic fields on the originally isothermal martensitic transformation also consists of cumulative three ones, magnetostatic, magnetic high field susceptibility and forced volume magnetostriction effects, as in the athermal martensitic transformations examined so far⁽⁹⁾⁽¹³⁾. That is, eq. (1) holds for both the kinetics of martensitic transformations. This suggests that difference between

the athermal and isothermal kinetics is not intrinsic but can be explained by one systematic consideration, which will be described in a separate paper⁽²⁰⁾.

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