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ARTICLE

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ABSTRACT

We have studied the structure change of an ordered Fe₃Pt (degree of order ~0.75) under a compressive stress applied in the [001] direction by neutron diffraction. In the absence of the stress, the alloy exhibits a weak first order martensitic transformation at 90 K from the $L1_2$ -type cubic structure to the $L6_0$ -type tetragonal structure. Under the compressive stress of 100 MPa, the first order nature of the thermally-induced martensitic transformation was undetectable in the temperature range of between 70 K and 270 K. The first order nature of the stress-induced martensitic transformation was also undetectable in the stress range of between 6 MPa and 300 MPa when tested at 120 K and higher temperatures. Under these conditions, the lattice parameters change continuously both in the cooling process and in the stress-applying process. Despite the disappearance of the first order nature of martensitic transformation, a significant stress-induced softening of lattice, which is regarded as a precursor phenomenon of martensitic transformation, was observed between 120 K and 265 K but not at 93 K and 295 K.

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I. INTRODUCTION

Martensitic transformations (MTs) are usually accompanied by shape change,¹ which is caused by the change in lattice parameters associated with the MTs.² Since the shape of stressstrain curves of shape memory alloys depends significantly on lattice parameters of the parent and martensite phases, the influence of external stress on lattice parameters is of fundamental importance to understand mechanical properties of shape memory alloys.

The MTs in representative shape memory alloys, such as Ti-Ni alloys,^{3,4} Cu-based alloys,^{5–7} and Ni-Mn-Ga alloys,^{8,9} are characterized by a remarkable discontinuity of lattice parameters. In these alloys, thermally-induced and stress-induced MTs both proceed by the coexistence of the parent and the martensite

phases with different lattice parameters. The strain-temperature curve under a fixed stress exhibits a sharp change in strain at the transformation temperature,^{10,11} and the stress-strain curve at a fixed temperature exhibits a plateau at the stress where the MT starts.⁵ The difference in strain before and after the transformation is termed as the transformation strain, $\Delta \varepsilon$, which is essentially determined by the lattice parameters of the parent and the martensite phases.¹² In these alloys, the martensitic transformation start temperature *T* increases almost linearly as stress σ increases. In a rough approximation, the slope is given by the following Clausius-Clapeyron equation:^{13,14}

$$\frac{d\sigma}{dT} = -\frac{\Delta S}{\Delta \varepsilon}.$$
(1)

Here, ΔS is the entropy change of the MT. Equation (1) determines the phase boundary between the parent and the martensite phases in the stress-temperature phase diagram. This phase boundary usually exists up to the yield stress for slip deformation. Since it is impossible to apply higher stress than the yield stress, Eq. (1) can be applied up to the yield stress. The entropy change ΔS and the transformation strain $\Delta \varepsilon$ in Eq. (1) generally depend on temperature and stress. However, in most shape memory alloys, the temperature and stress dependencies of these values are regarded to be small and are frequently treated as constants.

On the other hand, some alloys show significant temperature dependence of $\Delta \varepsilon$. It is reported in an Ni₅₀Fe₄₉Co₄Ga₂₇ (at. %) alloy that transformation strain $\Delta \varepsilon$ decreases significantly as test temperature increases.¹⁵ It is considered that the transformation strain of this alloy disappears completely, and the martensitic transformation may change to a continuous structure change at elevated temperatures. Similar disappearance of transformation strain by the application of compressive stress was observed in an Fe-31.2Pd alloy,¹⁶ which exhibits a weak first order MT from a cubic structure to a tetragonal structure. These results suggest that there is a critical point of martensitic transformation. We may regard that, when the stress or temperature is higher than the critical point, the first order nature of MT disappears and the MT becomes continuous. This kind of critical point is analogous to the critical point of vapor-liquid transformations. In a vaporliquid transformation, the volume change associated with the transformation disappears above the critical point, which corresponds to the disappearance of the transformation strain of a MT. The existence of the critical point of a MT was predicted previously by L'vov et al.¹⁷ using a Landau model.

We consider that alloys exhibiting a cubic-tetragonal MT are suitable to examine the critical point of MT under stress because in these alloys, the crystal structure of the martensite phase has the same symmetry as that of the parent phase under uniaxial stress. That is, the martensite phase is either contracted or expanded in the [001] direction compared with the parent phase, and such a structure can be induced by the application of uniaxial stress in the [001] direction of the parent phase. There are several alloys exhibiting cubic-tetragonal MT. In-Tl,^{18,19} Mn-Cu,^{20,21} Nb₃Sn,²² and V₃Si²³ are representative of alloys exhibiting cubic-tetragonal MT. Invar alloys, Fe-Pd^{24,25} and Fe-Pt,^{26,27} also exhibit cubic-tetragonal MT. In the present study, we focus on the so-called FCC (face centered cubic)-FCT (face centered tetragonal) MT in Fe₃Pt.

Martensitic transformation behavior of Fe_3Pt depends significantly on the degree of order of the parent phase with the $L1_2$ -type structure. There are at least four types of martensite phases.²⁸ The disordered alloy transforms to the body centered cubic martensite. As the degree of order increases, the structure of the martensite phase changes to the body centered tetragonal martensite. When further increasing the degree of order, it changes to the face centered tetragonal martensite with tetragonality less than 1 and then to the face centered tetragonal martensite with tetragonality larger than 1. Among these martensites, we are, in particular, interested in the face centered tetragonal martensite with tetragonality less than 1 because we can produce the tetragonal structure by the application of uniaxial compressive stress in the [001] direction of the parent phase. Although the structure of the parent phase is $L1_2$ -type and that of the martensite phase is $L6_0$ -type, we refer to this transformation as the FCC-FCT transformation in this paper following the conventional terminology.²⁹

The FCC-FCT MT in Fe₃Pt is associated with significant lattice softening.³⁰⁻³² The martensitic transformation temperature is approximately 90 K when the degree of order is 0.75.²⁸ According to our previous study,³³ the stress-strain curve tested just above the transformation temperature (90 K and 100 K) exhibited a plateau corresponding to the transformation strain characteristic to a first order martensitic transformation. However, the plateau was hard to identify when the test was made at 120 K and higher temperatures. Then, we suggested the existence of critical point for the FCC-FCT MT in Fe₃Pt.

In order to clarify the existence of critical point of the FCC-FCT MT in Fe₃Pt, we need to prove that the lattice parameters change continuously under compressive stress and that the strain induced by the stress application is caused by lattice strain, not by the change in the fraction of two phases with different lattice parameters. We partly reported that the stress-strain curve of Fe₃Pt at 93 K can be explained by the change in lattice parameters.³⁴ In this paper, we report the stress dependence of lattice parameters at higher temperatures. We also report temperature dependence of lattice parameters at a fixed compressive stress of 100 MPa. Based on these results, we discuss the existence of critical point of the FCC-FCT MT in Fe₃Pt.

II. EXPERIMENTAL PROCEDURE

A single crystal of Fe₃Pt was prepared by a floating zone method by using an ingot prepared by arc melting. A parallelepiped specimen with dimensions of $2.5 \times 2.6 \times 9.8 \text{ mm}^3$ was cut from the crystal for neutron experiments under compressive stresses. The compressive direction was [001] and the other two edges were [110] and $[1\overline{1}0]$ directions. The specimen was homogenized at 1373 K for 24 h followed by ordering heat-treatment at 923 K for 100 h. The degree of order of the L12-type structure after this heat-treatment was reported to be 0.75.²⁸ The martensitic transformation behavior in the absence of external stress was examined by magnetization measurements using a MPMS system of quantum design. Neutron diffraction experiments were made using a time of flight method at BL19 (TAKUMI) of J-PARC. The compressive axis was 45° from the incident beam as shown in Fig. 1. The lowest stress applied to the specimen was 6 MPa, which was needed to hold the specimen. Measurements were made in the stress-applying process after setting the test temperature to



FIG. 1. Experimental setup for neutron diffraction under compressive stress.

the lowest stress of 6 MPa. The diffraction profiles as a function of *d*-value in the compressive direction were detected by the $+90^{\circ}$ detector, and those perpendicular to the compressive direction were detected by the -90° detector. The instrumental peak width was tuned to 0.3%. Details are described in a previous paper.³⁴ We also made compressive tests by attaching a strain gage on the surface of the specimen.

III. RESULTS

Figure 2 shows temperature dependence of magnetic susceptibility of the present specimen measured in the cooling process. During the measurement, a weak magnetic field of 0.05 T was applied in the [001] direction. This direction is the hard axis of magnetization of the FCT martensite variant whose *c*-axis is perpendicular to the magnetic field. The magnetic susceptibility shows a very sharp decrease at 90 K. The sharp decrease in magnetization implies that first order martensitic transformation occurred at 90 K because of the following reasons: (i) since the applied magnetic field is weak, this sudden decrease in susceptibility implies sudden increase in magnetic anisotropy; (ii) the martensite phase of Fe₃Pt has a high magnetic anisotropy compared to the parent phase.

Figure 3 shows the intensity profile of neutron diffraction measured in a successive stress-applying process at 120 K. This temperature is by 30 K above the martensitic transformation temperature. We detected a clear peak of the (002) reflection by the +90° detector as shown in (a) and a peak of (220) reflection by the -90° detector as shown in (b). The interplanar distance (*d*-value) in the compressive direction (d_{002}) decreases and that perpendicular to it (d_{220}) increases as the compressive stress increases. We notice that both the (002) and the (220) peaks broaden as stress increases. Similar results were obtained at different examined temperatures (93 K, 190 K, 265 K, and 295 K). We evaluated the value of d_{220} and d_{002} by fitting each peak using a Lorentzian function. Then from these values, we calculated lattice parameters as $a = 2\sqrt{2}d_{220}$



FIG. 2. Magnetic susceptibility measured in the cooling process under a magnetic field of 0.05 T applied in the [001] direction.



FIG. 3. Neutron diffraction profiles obtained by TAKUMI. Peak profiles at 120 K under different compressive stresses (6, 90, 150, 240, and 300 MPa) detected by +90° detector (a) and those by -90° detector (b).

and $c = 2d_{002}$. The stress dependencies of the lattice parameters at the examined temperatures are shown in Fig. 4.

We know from Fig. 4 that *c* almost coincides with *a* at the lowest stress (6 MPa) except at 93 K. This implies that the structure is cubic at zero stress except at 93 K. The temperature of 93 K is very close to the martensitic transformation temperature; therefore, the martensite phase could be induced by the application of the low stress of 6 MPa. In fact, in a previous work,³³ we detected stress-induced martensitic transformation at 90 K under a low stress of approximately 2 MPa. That is, the stress-strain curve examined in the vicinity of martensitic transformation temperature exhibited a plateau of about 0.2% at the stress of approximately 2 MPa applied in the [001] direction. Therefore, we may regard that the specimen is in the FCT martensite state at 93 K in the present experimental condition: the compressive stress being higher than 6 MPa. As the stress increases, the lattice parameter *c* decreases and *a* increases monotonically at any temperature examined.

The lower panels in Fig. 4 show full width at half maximum (FWHM) of the (002) peak divided by d_{002} . The value of FWHM/ d_{002} is almost independent of stress at 190 K and higher temperatures. On the other hand, it increases significantly as stress increases at 93 K and 120 K. The value of FWHM/ d_{002} reaches about 5%. That is, lattice parameter is fluctuated by 5% in the specimen at these temperatures. This fluctuation in the lattice parameter is the same order as the lattice strain (see Fig. 5) under the compressive stress. We notice that FWHM/ d_{002} tends to be large when the lattice strain is large, but the precise relation is yet to be obtained. One possibility of the large FWHM under large strain could be the constraint of the specimen by pistons, which apply stress to the specimen.

Figure 5 compares lattice strain obtained by neutron experiment (red circles) with the elastic strain measured by attaching a strain gage on the surface of the specimen (solid lines). The lattice strain was evaluated as $(c - c_0)/c_0$, where c_0 is the extrapolated lattice parameter *c* to zero stress at each temperature. The lattice strain almost coincides with the elastic strain. The small deviation



FIG. 4. Stress dependence of lattice parameters (upper panels) and FWHM (lower panels) of the 002 reflection. Measurements were made at 93, 120, 190, 265, and 295 K.

between them is probably caused by inaccuracy of temperature control. The red curves in Fig. 5 are the fitted curves using second-order polynomials. The agreement between the lattice strain and elastic strain implies that the elastic strain is essentially caused by the lattice strain. It is not caused by the change in the fraction of two phases with different lattice parameters unlike typical shape memory alloys.

We also evaluated temperature dependence of lattice parameters *a* and *c* under a fixed compressive stress of 100 MPa. The result is shown in Fig. 6 together with the lattice parameters under zero stress reported in a previous paper.³⁵ Both the lattice parameters *a* and *c* change gradually as temperature decreases. Incidentally, the lack of data in the vicinity of 180 K is due to the lack of neutron beam during the experiments; it is not purposely removed.

IV. DISCUSSION

The martensitic transformation under zero stress can be clearly detected by magnetic susceptibility as observed in Fig. 2.

The transformation temperature under zero stress can also be known from the temperature dependence of lattice parameters as shown in Fig. 6. On the other hand, the transformation temperature under compressive stress of 100 MPa is not clear. As seen in Fig. 6, both *a* and *c* change continuously as temperature decreases. We cannot specify the transformation temperature under 100 MPa from the temperature dependence of lattice parameters. We may consider that the martensitic transformation temperature disappears under the compressive stress. In other words, the FCT structure of the Fe₃Pt evolves continuously in the cooling process when a fixed compressive stress is applied in the [001] direction. This behavior is analogous to the disappearance of Curie temperature of paramagnetic to ferromagnetic transition under a magnetic field. That is, the magnetization under a magnetic field increases continuously as temperature decreases and the Curie temperature cannot be detected. This behavior is also analogous to the disappearance of vapor-liquid transformation above its critical point.

Similar evolution of the FCT structure can be seen in the stress-applying process at a fixed temperature of 120 K. At this



FIG. 5. Lattice strain (solid circles) evaluated from lattice parameter *c*, and elastic strain (solid black lines) evaluated by strain gage. Red lines are fitted curves using second-order polynomials.



FIG. 6. Temperature dependence of lattice parameters under 100 MPa applied in the [001] direction. The lattice parameters under zero stress are also shown for comparison.

temperature, *c* and *a* are the same at zero stress. That is, the structure is cubic under zero stress. As stress increases, both *c* and *a* change gradually, and the tetragonality (*c/a*) under 300 MPa is 0.925. This value is smaller than c/a of the FCT martensite at 14 K (0.945). Therefore, we may regard that the phase at 120 K under 300 MPa is the FCT martensite, although no sign of clear martensitic transformation was detected in the stress-applying process.

Although the structure change under compressive stress is continuous as discussed above, the relation between stress and lattice parameters is not linear as seen in Fig. 4. The nonlinearity is also seen in the lattice strain and elastic strain shown in Fig. 5. In order to evaluate the nonlinearity, we fitted the relation between the lattice strain (ε) and stress (σ) by using a second-order polynomial

$$\sigma = E_1 \varepsilon + E_2 \varepsilon^2. \tag{2}$$

The fitted results are shown by red curves in Fig. 4. The fitting parameters E_1 and E_2 are summarized in Fig. 7. The parameter E_1 , which corresponds to Young's modulus, decreases almost linearly as temperature decreases. The parameter E_2 shows a rather complex behavior. It is negative except at 295 K. The negative value of E_2 implies that softening occurs by stress application. The stress-induced softening is especially significant at 190 K. In fact, we notice in Fig. 5 that the slope of the stress-strain curve changes significantly at 190 K. The relation between stress-induced softening and martensitic transformation was discussed previously by Li *et al.*,³⁶ Ding *et al.*,³⁷ and Seiner *et al.*³⁸ In Ref. 36, influence of softening of Young's modulus in the $\langle 111 \rangle$ direction on the B2-R transformation in Ti-Ni alloys was demonstrated. In Ref. 37, stress-induced softening of C' prior to martensitic transformation was demonstrated by a molecular dynamics simulation. In Ref. 38, stress dependence of elastic constants of Fe-31.2Pd, which exhibits FCC-FCT transformation was examined; it was found that elastic anisotropy (C_{44}/C') changes from 24.3 to 52 by the application of compressive stress of 200 MPa in the [001] direction due to



FIG. 7. Temperature dependence of elastic moduli E_1 and E_2 .

softening of C' while hardening of C_{44} . The small value of E_2 at 93 K and 120 K will be due to the fact that Young's modulus E_1 is sufficiently small at these temperatures; therefore, further softening by stress application is limited.

It is well known that the Invar effect of Fe₃Pt is strong. Its volume increases as temperature decreases.³⁹ Therefore, it is of interest to examine how the volume of unit cell changes by the application of compressive stress in the Invar region. Figure 8 summarizes the volume of unit cell ($V = ca^2$) as a function of applied stress. We first notice in Fig. 8 that the volume of the unit cell under zero stress increases as temperature decreases as expected from the Invar effect of this alloy. The average thermal expansion coefficient between 295 K and 190 K is -1.2×10^{-5} /K.



FIG. 8. Volume of unit cell plotted as a function of compressive stress.

At 295 K, 265 K, and 190 K, V decreases monotonically as stress increases. This is the conventional behavior of most metals and alloys. At 120 K, however, V shows a minimum near 200 MPa. Above 200 MPa, the volume of the unit cell increases as compressive stress increases. This implies that Poisson's ratio exceeds 0.5 at 120 K when the stress exceeds 200 MPa. Poisson's ratio never exceeds 0.5 in isotropic materials according to the elastic theory. However, the present specimen is anisotropic because it is a single crystal, and the elastic anisotropy, which is defined as the ratio of two elastic constants C_{44}/C' , is higher than 10 at 120 K as reported before.³² Such a high elastic anisotropy could be one reason for the increase of volume under compressive stress. Another possible reason for the increase of volume by the increase of compressive stress is the increase in spontaneous magnetization. In Invar alloys, interaction between magnetic moments is considered to be the cause of negative thermal expansion. In Fe₃Pt, the spontaneous magnetization with different c/a was examined by using four types of martensite phases,²⁸ and it was confirmed that the spontaneous magnetization at a fixed temperature increases as c/a decreases. Therefore, it is expected that spontaneous magnetization of the present specimen increases as stress increases, which may cause volume expansion under compressive stress. Further experiments are necessary to confirm this explanation. Incidentally, the stress dependence of V at 93 K is shown only for the region of up to 140 MPa because the calculated values above this stress largely scattered and was not reliable. Presumably, the large value of FWHM of the diffraction pattern is the reason for the scattering. Therefore, we are not sure whether the volume expansion observed at 120 MPa also appears at 93 K or not. Our recent preliminary experiment on an Fe-Pd alloy suggests that this alloy also exhibits volume expansion by the application of compressive stress in the [001] direction. Experimental results of the Fe-Pd alloy will be presented in a separate paper.

V. CONCLUSIONS

Partly ordered Fe₃Pt with a degree of order ~0.75 exhibits weak first order martensitic transformation in the absence of external stress. The transformation is detected as a sharp decrease in the magnetic susceptibility due to the sudden increase of magnetic susceptibility. Under the compressive stress applied in the [001] direction, however, the first order nature of the transformation disappears. The structure continuously changes in the cooling process and also in the stress-applying process. The disappearance of the first order nature of the FCC-FCT martensitic transformation implies the existence of a critical point like that in vapor-liquid transformations. Although the first order nature of martensitic transformation disappears under compressive stress, stress-induced softening occurs in the specimen, which is possibly related to precursor phenomena of martensitic transformation of this alloy. Volume expansion under compressive stress was also observed.

SUPPLEMENTARY MATERIAL

See the supplementary material for stress-strain curves with a plateau of stress-induced martensitic transformation at 90 K and 100 K, and temperature dependence of the neutron diffraction profile measured under 100 MPa.

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