

# The Origin of Midrib in Lenticular Martensite

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In the present paper, the origin of midrib in lenticular martensite is clarified by examining the similarity between midrib and thin plate martensite in detail and studying the stress-induced growth behavior of thin plate martensite at various temperatures. Although lenticular martensite, especially midrib, exhibits a zigzag array in general, some martensite plates which are branched or kinked were also observed as thin plate martensite. The substructure of midrib is completely twinned and the orientation relationship of midrib with respect to austenite is close to Greninger–Troiano relationship. These morphology, substructure and crystallographic features of midrib in lenticular martensite are quite similar to those of thin plate martensite. Furthermore, stress-induced growth behavior of thin plate martensite changes with deformation temperature. Thermally-transformed thin plate martensite grows keeping a thin plate shape when deformed at temperature close to the Ms temperature. However, it grows into a lenticular shape accompanying a substructure with a high density of dislocations after deformation at temperature much higher than Ms temperature. Therefore, it is concluded that midrib in lenticular martensite is thin plate martensite itself. The difference between lenticular martensite and thin plate martensite is only in their growth behaviors. [doi:10.2320/matertrans.MRA2007296]

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## 1. Introduction

In ferrous  $\alpha'$  martensite (bcc or bct structure), there are several different morphologies, *i.e.*, lath, butterfly, lenticular and thin plate, each of which is unique in crystallographic features as well as in substructure.<sup>1)</sup> Among these ferrous  $\alpha'$  martensite, the substructure of lenticular martensite is much more complicated than that of other morphologies and it consists of the following three distinctive regions: midrib, twinned region and untwinned region.<sup>2,3)</sup> The midrib is contained in the middle of lenticular martensite plate. Although it is well known that midrib is the region in which high density of transformation twins exist,<sup>2-5)</sup> the origin and nature of midrib has not been clear yet. However, it is considered that the formation of lenticular martensite is triggered by the formation of midrib.

There are some experimental evidences supporting this view. Neuhäuser and Pitch<sup>6)</sup> studied the effect of incoherent precipitate particle ( $\text{Fe}_3\text{P}$  or  $\text{Ni}_3\text{P}$ ) on the martensitic transformation in an Fe-31Ni-1P alloy and showed that small austenite is retained around the incoherent precipitate particle in martensite. Since the shapes of these retained austenite are opposite on each side of midrib, they proposed that midrib is the plane from which martensitic transformation starts. Kakeshita *et al.*<sup>7)</sup> studied the growth behavior of lenticular martensite in an Fe-30Ni-0.4C alloy by repeating subzero-cooling and microstructure observation. They reported that midrib, which is thin plate shape,

appears in the earliest stage of martensitic transformation and martensite morphology becomes lenticular by further growth. Once lenticular martensite plate reaches the final size, the martensite/austenite interface (M/A interface) never grows further.

Since the M/A interface of lenticular martensite is smoothly curved and does not have a definite habit plane, midrib plane is usually taken as a habit plane, which is reported to be close to  $\{259\}_A$  or  $\{31015\}_A$  (hereafter subscripts 'A' and 'M' represent austenite and martensite, respectively).<sup>8-10)</sup> This plane is consistent with the invariant plane calculated from phenomenological theory of martensite crystallography.<sup>11-14)</sup>

As described above, midrib of lenticular martensite is (1) the region which contains high density of transformation twins, (2) the place where martensitic transformation starts, (3) thin plate shape whose orientation is close to  $\{259\}_A$  or  $\{31015\}_A$ .

Thin plate martensite, which forms in a lower temperature range than that of lenticular martensite, has a planar M/A interface and contains a set of regularly spaced transformation twins extending from one M/A interface to the other.<sup>15)</sup> The habit plane is reported to be close to  $\{31015\}_A$ .<sup>16,17)</sup> Among various  $\alpha'$  martensite morphology, only thin plate martensite has a mobile M/A interface and exhibits a shape memory effect.<sup>18-20)</sup>

As mentioned above, basic characters of lenticular martensite and thin plate martensite are completely different. However, when only midrib is paid attention, substructures and habit planes of midrib and thin plate martensite are quite similar.

In the present study, the origin of midrib was clarified by examining the similarity between midrib and thin plate martensite in detail and studying the stress-induced growth behavior of thin plate martensite at various temperatures.

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Table 1 Chemical compositions (mass%) and Ms temperatures (K) of the alloys used in the present study.

Alloys	Ni	C	Co	Ti	Fe	Ms (K)	Morphology of $\alpha'$ martensite
Fe-29Ni-0.2C	28.94	0.22	—	—	bal.	198	lenticular
Fe-31Ni	30.76	—	—	—	bal.	223	lenticular
Fe-33Ni	32.85	—	—	—	bal.	171	lenticular
Fe-31Ni-10Co-3Ti	31.13	—	9.96	3.04	bal.	83	thin plate

## 2. Experimental Procedure

The materials used in the present study are listed in Table 1. Specimens were austenitized at 1473 K for 3.6 ks (Fe-31Ni, Fe-33Ni and Fe-31Ni-10Co-3Ti) or 1273 K for 3.6 ks (Fe-29Ni-0.2C) and quenched into water. The as-quenched specimens were fully austenitic at room temperature. Fe-29Ni-0.2C, Fe-31Ni and Fe-33Ni alloys were cooled just below the Ms temperature (198 K, 223 K and 171 K, respectively) to obtain the microstructure in which lenticular martensite is partly formed. An Fe-31Ni-10Co-3Ti alloy was cooled just below the Ms temperature (83 K) to obtain thin plate martensite, and subsequently deformed in tension at temperatures between 77 K and 200 K in order to observe the stress-induced growth behavior.

Microstructures were observed by means of optical microscopy and TEM (Philips: CM200 and CM200FEG). For optical microscopy observation, specimens were etched by 5% nital (Fe-29Ni-0.2C) or a mixture of 35 g  $\text{Na}_2\text{S}_2\text{O}_5$  + 65 mL  $\text{H}_2\text{O}$  (Fe-31Ni-10Co-3Ti) after mechanical and electrolytic polishing using a solution of 500 mL  $\text{CH}_3\text{COOH}$  + 20 mL  $\text{H}_2\text{O}$  + 100 g  $\text{CrO}_3$  at 283 K. Thin-foil specimens for TEM observation were prepared by twin-jet electrolytic polishing at 283 K using the same solution as optical microscopy observation.

Orientation relationship of martensite with respect to austenite was measured with electron back scattering diffraction (EBSD). EBSD measurements were conducted by using SEM (Jeol: JSM6500F) and EBSD analysis was performed with the TSL OIM system.

## 3. Results and Discussion

### 3.1 Morphology

Optical micrographs in Fig. 1 show the typical morphologies of (a) lenticular martensite in an Fe-29Ni-0.2C alloy and (b) thin plate martensite in an Fe-31Ni-10Co-3Ti alloy, respectively. In Fig. 1(a), the M/A interface of lenticular martensite is smoothly curved and midrib can be recognized by etching. Lenticular martensite forms in a group and exhibits a zigzag array. These arrays are quite similar to those of thin plate martensite as shown in Fig. 1(b). These martensite plates consist of several variants which are self-accommodating of transformation strain one another.<sup>21,22)</sup>

All lenticular martensite plate do not necessarily exhibit such a typical morphology and some irregular morphologies can be observed. Figures 2(a), (c) and (e) show the various kinds of irregular morphologies of lenticular martensite. Midrib in lenticular martensite is sometimes branched (Fig. 2(a)) or kinked (Fig. 2(c)) as indicated by the arrows,

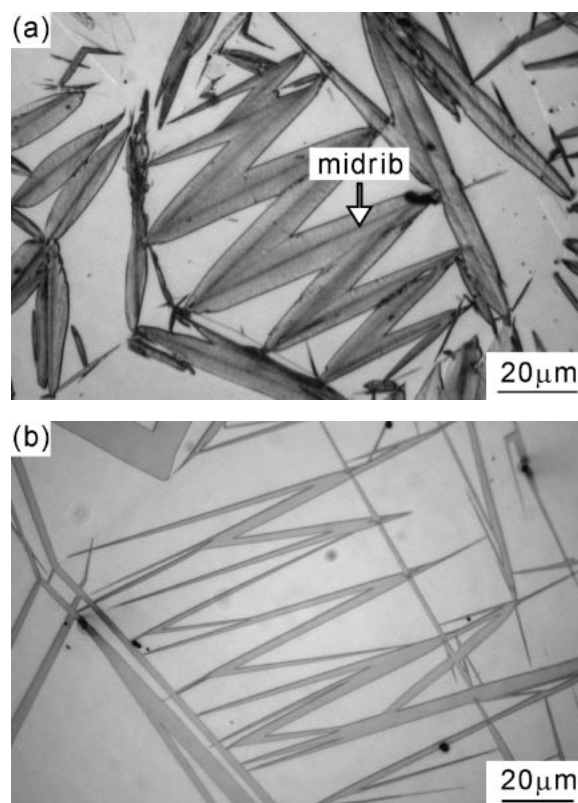


Fig. 1 Optical micrographs showing (a) lenticular martensite in Fe-29Ni-0.2C ( $M_s = 198$  K), (b) thin plate martensite in Fe-31Ni-10Co-3Ti ( $M_s = 83$  K), respectively.

respectively. These kinds of morphology such as branch or kink are often observed in thin plate martensite (Figs. 2(b) and (d)) as previously reported by Maki *et al.*<sup>23)</sup> One midrib is not necessarily exist in one lenticular martensite plate and more than two midribs may exist as shown in Fig. 2(e) as previously reported.<sup>24,25)</sup> This configuration of midrib is quite similar to the morphology of thin plate martensite shown in Fig. 2(f). In this case, some martensite plates with same variant form closely one another.

From these results, the morphology of midrib in lenticular martensite is almost the same as that of thin plate martensite.

### 3.2 Substructure

Lenticular martensite consists of three distinctive regions in terms of substructure: midrib, twinned region and untwinned region.<sup>2,3)</sup> Figure 3(a) is a TEM micrograph showing the substructure of midrib in lenticular martensite in an Fe-31Ni alloy and (b), (c) are corresponding diffraction pattern and its key diagram, respectively. Midrib region

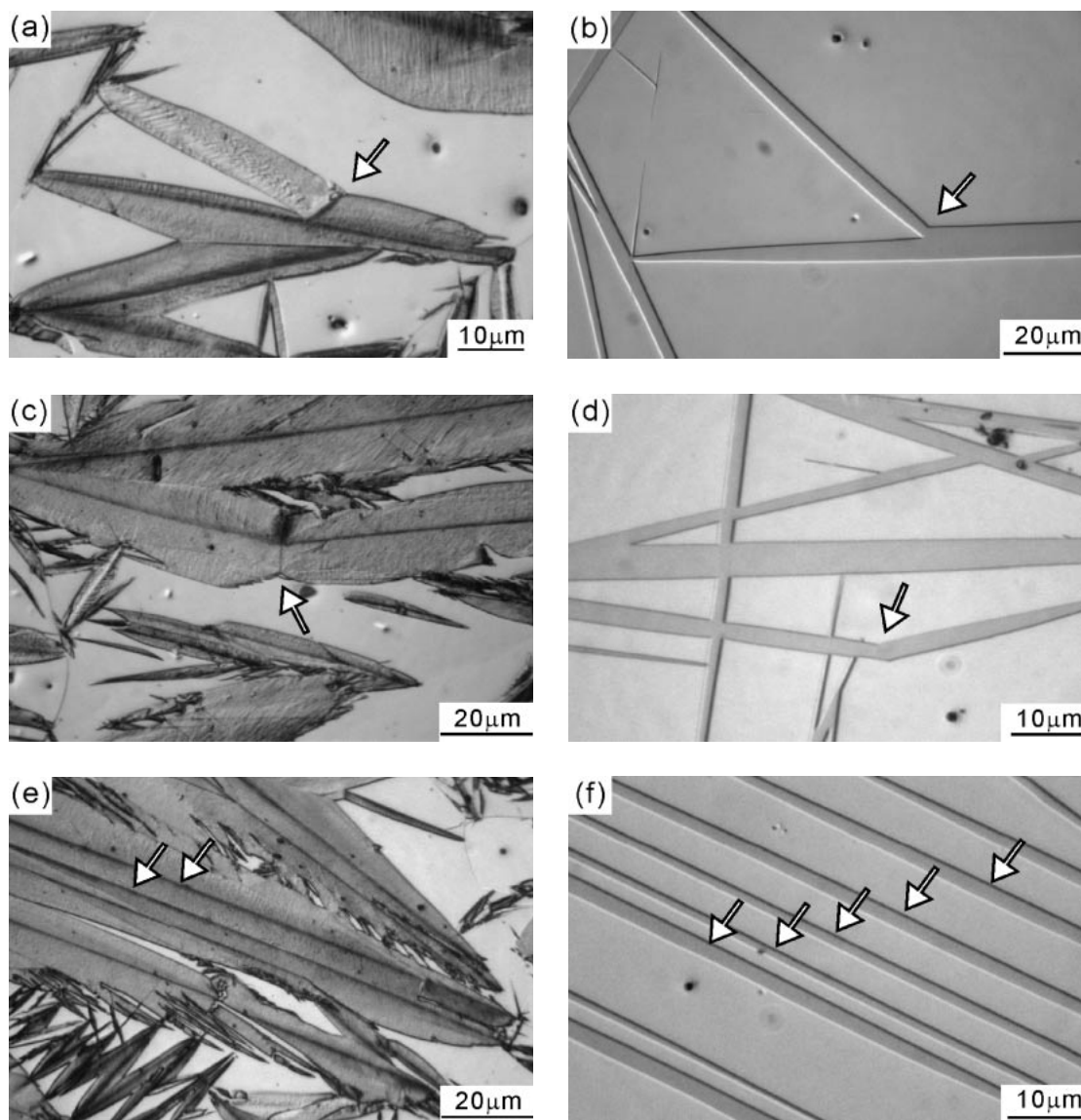


Fig. 2 Optical micrographs showing lenticular martensite in Fe-29Ni-0.2C ( $M_s = 198$  K) (a, c, e) and thin plate martensite in Fe-31Ni-10Co-3Ti ( $M_s = 83$  K) (b, d, f).

contains fine transformation twins, whose width is about 6.9 nm. From diffraction pattern and trace analysis, transformation twin plane is  $(112)_M$ . Figure 4(a) is a TEM micrograph showing the substructure of thin plate martensite in an Fe-31Ni-10Co-3Ti alloy and (b), (c) are corresponding diffraction pattern and its key diagram. Thin plate martensite also contains fine transformation twins which extend from one M/A interface to the other and its twin system is the same as that of midrib. Furthermore, almost no dislocations are observed in the surrounding austenite. The twin width of thin plate martensite in Fig. 4(a) is about 6.4 nm and a little narrower than that of midrib in Fig. 3(a). It was reported that the width of transformation twin decreases with an increase in tetragonality of martensite.<sup>26,27</sup> Therefore, the twin width in an Fe-31Ni-10Co-3Ti alloy is narrow because of its high tetragonality ( $c/a \sim 1.03^{28}$ ). However, the fractions of transformation twin of thin plate martensite and midrib in lenticular martensite are 0.33 and 0.35, respectively, and almost the same.

Although the substructure of lenticular martensite changes from twins to dislocations as approaching M/A interface, the substructure of midrib, which consists of fine transformation twins, is completely the same as that of thin plate martensite.

### 3.3 Crystallographic features

Figure 5(a), (b) show the  $[\bar{1}\bar{1}1]_M$  directions of midrib region in an Fe-33Ni alloy and thin plate martensite in an Fe-31Ni-10Co-3Ti alloy, respectively, plotted on the  $[001]_A$  stereographic projection (enlarged region around  $[\bar{1}01]_A$ ). These points express experimental data collected from many different martensite plates. The ideal  $[\bar{1}\bar{1}1]_M$  positions for Kurdjumov–Sachs (K-S) relationship ( $(111)_A // (011)_M$ ,  $[\bar{1}01]_A // [\bar{1}\bar{1}1]_M$ ) and Nishiyama–Wassermann (N-W) relationship ( $(111)_A // (011)_M$ ,  $[\bar{1}\bar{1}2]_A // [01\bar{1}]_M$ ) are also plotted. The  $[\bar{1}\bar{1}1]_M$  position calculated using the phenomenological theory of martensite crystallography (PTMC)<sup>11,14</sup> is represented as solid star symbol. The PTMC calculation was performed by using the lattice parameters of austenite

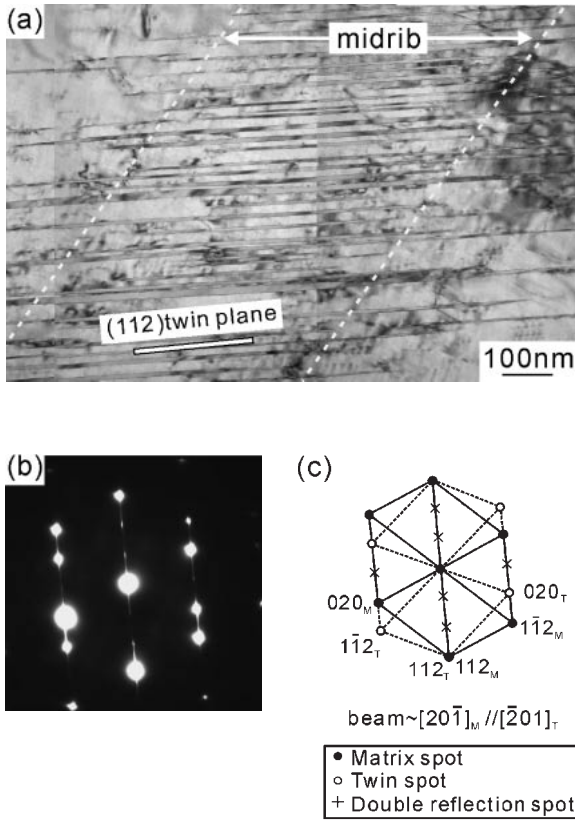


Fig. 3 TEM micrographs showing the substructure of midrib in lenticular martensite in Fe-31Ni ( $M_s = 223$  K): (a) bright field image, (b) corresponding diffraction pattern, (c) key diagram of (b) (subscripts 'M' and 'T' represent martensite and transformation twin of martensite, respectively).

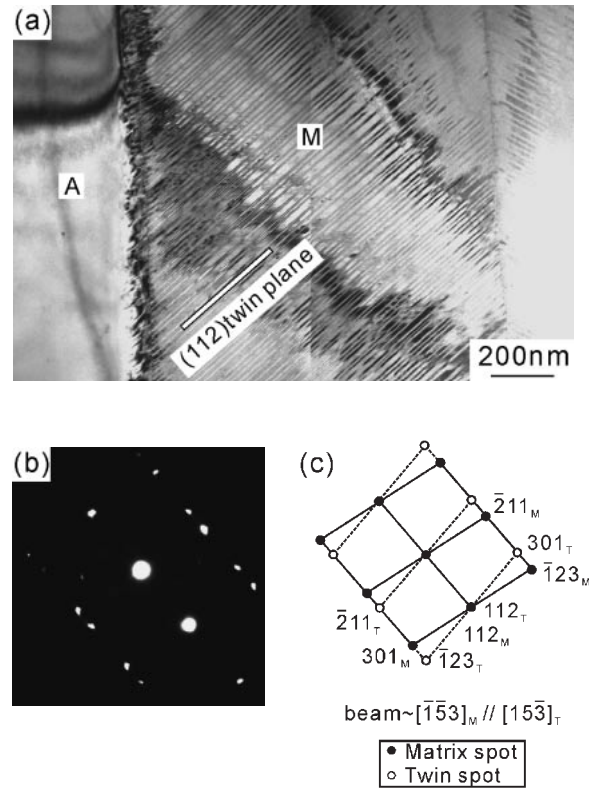


Fig. 4 TEM micrographs showing the substructure of thin plate martensite in Fe-31Ni-10Co-3Ti ( $M_s = 83$  K): (a) bright field image, (b) corresponding diffraction pattern, (c) key diagram of (b) (subscripts 'M' and 'T' represent martensite and transformation twin of martensite, respectively).

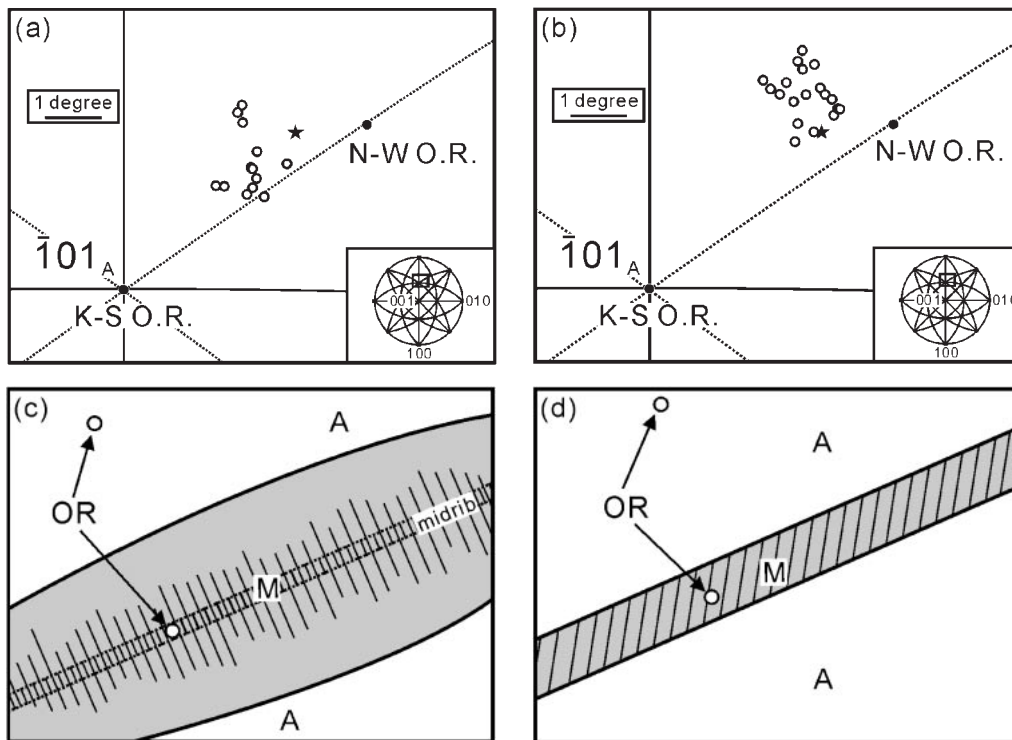


Fig. 5 (a), (b) Stereographic projections around  $[\bar{1}01]_A$  axis showing the orientation relationship of midrib in lenticular martensite in Fe-33Ni ( $M_s = 171$  K) and thin plate martensite in Fe-31Ni-10Co-3Ti ( $M_s = 83$  K), respectively. Open circles show the collected  $[\bar{1}\bar{1}1]_M$  directions obtained from many different martensite plates and the ideal  $[\bar{1}\bar{1}1]_M$  directions of K-S relationship and N-W relationship are shown as solid circles. The solid stars represent the calculated  $[\bar{1}\bar{1}1]_M$  direction using PTMC. (c), (d) Schematic illustrations showing measured points of orientation of martensite and austenite in (a) and (b), respectively.

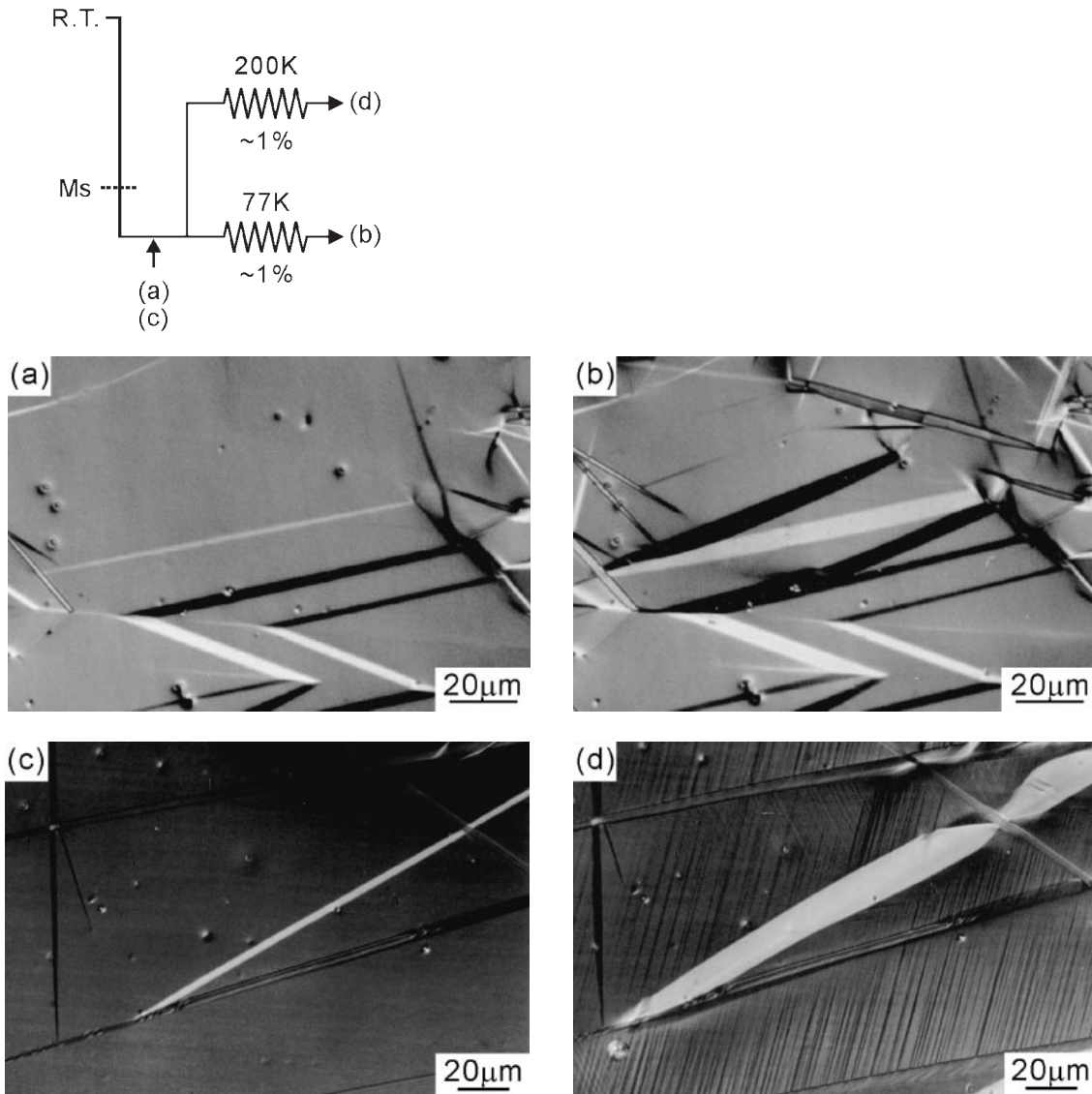


Fig. 6 Series of optical micrographs (surface relieves) showing the stress-induced growth behavior of thin plate martensite by tensile deformation in Fe-31Ni-10Co-3Ti ( $M_s = 83$  K): (a), (c) thin plate martensite formed by subzero cooling at 77 K, (b) same martensite plates as (a) after about 1% tensile deformation at 77 K, (d) same martensite plates as (c) after about 1% tensile deformation at 200 K.

and martensite in an Fe-33Ni alloy ( $a_A = 0.3590$  nm,  $a_M = 0.2854$  nm)<sup>3)</sup> and assuming the  $(112)_M$  deformation twin as lattice invariant deformation.

In order to measure the orientation relationship of lenticular martensite with respect to austenite, the orientation change in the surrounding austenite should be taken into consideration as previously reported.<sup>3)</sup> It is assumed that surrounding austenite is not plastically deformed when midrib region is formed because austenite surrounding thin plate martensite is usually not plastically deformed as shown in Fig. 4. Thus, the orientation relationship for midrib region was measured from the orientations of midrib region and austenite far from the M/A interface (more than  $15 \mu\text{m}$  away from the M/A interface), which is not deformed by the formation of lenticular martensite, as shown schematically in Fig. 5(c). Although the surrounding austenite of thin plate martensite is not deformed, the orientation relationship of thin plate martensite was also taken against the austenite far from the M/A interface (Fig. 5(d)).

In Fig. 5(a),  $[\bar{1}\bar{1}1]_M$  directions of midrib are distributed in the middle of K-S and N-W, indicating that orientation relationship is near Greninger-Troiano (G-T) relationship ( $(111)_A$   $1^\circ$  from  $(011)_M$ ,  $[\bar{1}01]_A$   $2.5^\circ$  from  $[\bar{1}\bar{1}1]_M$ ). Although the orientation relationship of thin plate martensite is deviated by about  $1^\circ$  from that of midrib as shown in Fig. 5(b), it can be said that orientation relationships of midrib and thin plate martensite are nearly the same.

Since the habit plane of lenticular martensite (that is, midrib plane) and thin plate martensite are also close each other,<sup>8-10,16,17)</sup> it is concluded that crystallographic features of midrib are also almost the same as those of thin plate martensite.

#### 3.4 Stress-induced growth behavior of thin plate martensite

As described above, midrib in lenticular martensite is quite similar to thin plate martensite from view points of morphology, substructure and crystallographic features.

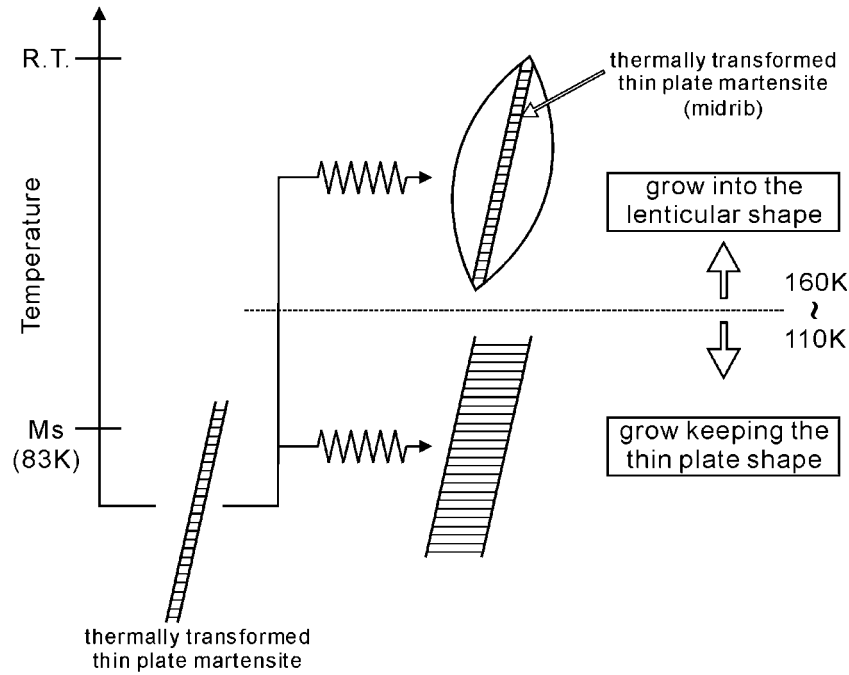


Fig. 7 Summary of the stress-induced growth behavior of thin plate martensite by tensile deformation at various temperatures in Fe-31Ni-10Co-3Ti ( $M_s = 83$  K).

Therefore, we concluded that midrib is thin plate martensite itself and that the difference between lenticular and thin plate martensites is only in their growth behaviors. In order to confirm this consideration, growth behavior of thin plate martensite by applying stress at various temperatures was examined.

Figure 6 is optical micrographs showing the stress-induced growth behavior of thin plate martensite by tensile deformation at 77 K and 200 K. As shown in Figs. 6(a) and (c), thin plate martensite, which can be observed as surface relieves, is formed by subzero cooling to 77 K. In Fig. 6(b), the thermally-transformed thin plate martensite thickens but still maintain a thin plate shape after 1% tensile deformation at 77 K. On the other hand, the thermally-transformed thin plate martensite grows into a lenticular shape after 1% tensile deformation at 200 K (about 120 K higher than  $M_s$  temperature) as shown in Fig. 6(d).

Figure 7 summarizes the stress-induced growth behavior of thin plate martensite by tensile deformation at various temperatures. The thermally-transformed thin plate martensite grows keeping a thin plate shape when deformed at below 110 K and grows into a lenticular shape at above 160 K. In the temperature range between 110 and 160 K, both martensite plates with a thin plate shape or lenticular shape are observed. Consequently, the stress-induced growth behavior of thin plate martensite changes with deformation temperature.

Figure 8(a) is a TEM micrograph showing the substructure of lenticular shaped martensite grown from thermally-transformed thin plate martensite by 1% tensile deformation at 160 K. In Fig. 8(a), there are two areas with different substructures ( $M_1$  and  $M_2$ ). Since the region denoted as  $M_1$  contains high density of fine transformation twins, this region corresponds to the thermally-transformed thin plate martensite. On the other hand, in the region of  $M_2$ , the density of

transformation twins is lower than that of  $M_1$  and dislocations are observed. Fig. 8(b) is a high magnification of TEM micrograph showing the region between  $M_1$  and  $M_2$  in Fig. 8(a). The transformation twins are partly extended from  $M_1$  region to  $M_2$ , indicating that  $M_2$  is the region formed by tensile deformation. It is considered that the lattice invariant deformation mode changes from twinning to slip due to the high deformation temperature (about 80 K higher than  $M_s$  temperature). Since the lenticular shaped martensite formed from thin plate martensite consists of the completely twinned region ( $M_1$ ) and partially twinned region ( $M_2$ ), the substructure of this martensite is quite similar to that of lenticular martensite.

These results clearly indicate that midrib in lenticular martensite is thin plate martensite itself. There is no difference between lenticular martensite (that is, midrib) and thin plate martensite at the earliest stage of martensitic transformation. Stress-induced growth behavior of thin plate martensite (or midrib) depends on temperature. When formation temperature is quite low, martensite can grow keeping a thin plate shape. On the other hand, when formation temperature is relatively high, the change in deformation mode from twinning to slip result in the growth of martensite into a lenticular shape. This change in lattice invariant deformation mode is due to the local temperature rise caused by latent heat as proposed by Patterson and Wayman.<sup>2)</sup> Although local temperature is also raised in the case of thin plate martensite, deformation mode remains twinning because of quite low formation temperature.

#### 4. Conclusion

- (1) Lenticular martensite, especially midrib, usually exhibits a zigzag array. However, some martensite plates which are branched or kinked were also observed. The

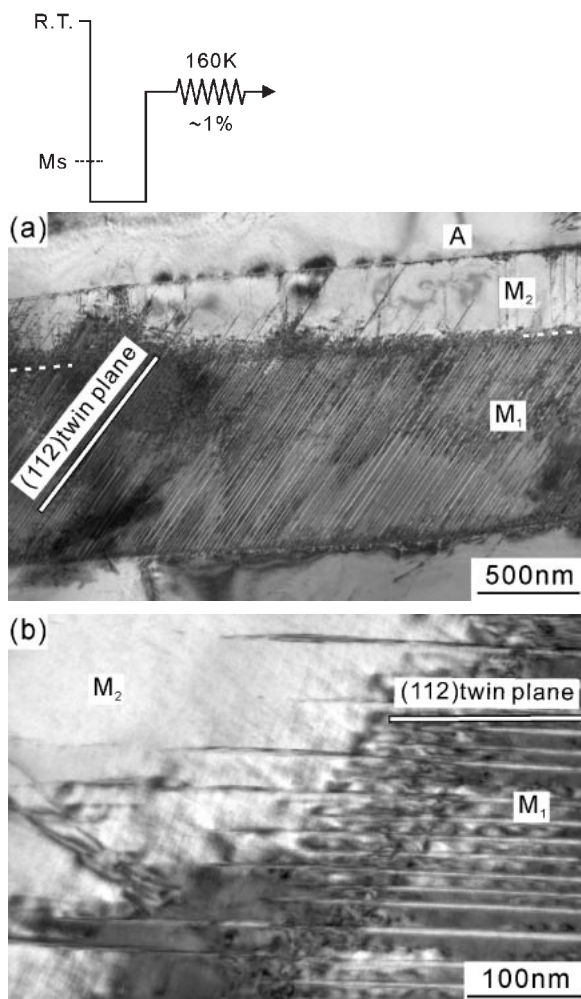


Fig. 8 (a) TEM micrograph showing the substructure of lenticular shaped martensite grown from thermally-transformed thin plate martensite by about 1% tensile deformation at 160 K in Fe-31Ni-10Co-3Ti ( $M_s = 83$  K) ( $M_1$ : the region of thermally-transformed thin plate martensite,  $M_2$ : the stress-induced growth region formed by tensile deformation, A: surrounding austenite) and (b) high magnification of the region between  $M_1$  and  $M_2$  in (a).

substructure of midrib is completely twinned and the orientation relationship of midrib with respect to austenite is close to G-T relationship. These morphology, substructure and crystallographic features of midrib in lenticular martensite are quite similar to those of thin plate martensite.

- (2) Stress-induced growth behavior of thin plate martensite changes with deformation temperature. Thermally-transformed thin plate martensite formed at  $M_s$  temperature (83 K) grows keeping a thin plate shape by tensile deformation below 110–160 K. On the other hand, thin plate martensite grows into a lenticular shape by tensile deformation above 110–160 K.
- (3) It is concluded that midrib in lenticular martensite is thin plate martensite itself. The difference between lenticular martensite and thin plate martensite is only

in their growth behaviors. In the case of lenticular martensite, the lattice invariant deformation mode changes from twinning to slip during growth after formation of midrib due to the local temperature rise in martensite. On the other hand, that of thin plate martensite is not changed during growth because of quite low formation temperature.

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