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New observations on the formation of strain-induced martensite in an Fe–29.6%Ni alloy

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Observations of the morphology and substructure of strain-induced martensites in an Fe–29.6%Ni alloy revealed the formation of butterfly- and lath-like transformation products with $\{11\mathrm{NJ}2\}_b$ $\langle 111\rangle_b$ type transformation twins and a parallel array of straight screw dislocations along $\langle 111\rangle_b$ directions depending upon the amount of plastic deformation. Crystallographic analysis showed the formation of strain-induced martensites with unique $\{225\}_f$ habit plane and a Kurdjumov–Sachs-type orientation relationship relative to the matrix austenite at least up to the large deformation ranges which could easily make the crystallographic measurements unfeasible. It has been shown that the strain-induced martensites are formed with more elongated shapes as the amount of plastic deformation is increased.

1. Introduction

A considerable number of studies have been reported on the morphology and crystallography of strain-induced martensites in different iron alloys [1–9]. Early electron microscope observations in some Fe–Ni alloys showed that the morphology of strain-induced martensite is affected by the amount of austenite deformation and it is different from that of the thermally induced martensites [1]. According to the reported results, strain-induced martensite in iron alloys is generally formed in needle-like shapes but butterfly (or chevron)-like morphologies may also be observed under different transformation conditions [1, 7, 8]. It is well established that the substructure of strain-induced martensite is very different from that of thermally induced martensite and contains very high densities of dislocations in the form of lattice imperfections. However, it was revealed in some Fe–Ni–C alloys after *in situ* electron microscope observations of the strain-induced martensite formation, that this type of martensite also contains transformation twins and the appearance of the twins depends on their orientation with respect to the specimen surface [10].

The reported results on the substructure of small martensite plates or needles have been obtained generally by transmission electron microscope studies. As it was noted by some authors, careful tilting of the electron microscope specimens is very important in the examination of twinned substructures [10, 11]. Tan *et al.* [11] explained the observed morphology and substructure changes due to tilting of thin electron microscope samples and showed that the image of the twins may vanish with very small deviations from the exact Bragg conditions in thermally induced lath martensites of some iron alloys. According to their conclusions, lath martensites may be formed with internal twins but could not be observed without proper tilt angles. They also noted the important effects of martensite plate size, orientation of twin planes relative to the specimen surface and the specimen thickness on the observation of martensite twins.

The purpose of the present study was to examine the formation of strain-induced martensite in an Fe–29.6% Ni alloy and determine the crystallographic parameters of the product structures which are formed during the austenite to strain-induced martensite transformation by using transmission electron microscope (TEM) techniques.

2. Experimental procedure

The Fe–29.6% Ni alloy was prepared by vacuum induction melting. Samples were austenized at 1200 °C in vacuum for 8 h and cooled to room temperature. The M_s temperature was measured as -40 °C. The austenitic bulk samples were deformed plastically by compression at room temperature to obtain strain induced martensite and the load cell of the compression test machine was calibrated to 1% prior to each test. A crosshead speed of 150 μm min^{-1} was used during all experiments. Thin foil samples used in TEM observations were prepared from 3 mm discs electropolished by using a double-jet polishing technique with a solution of methanol–25% perchloric acid at -5 °C. The specimens were examined in a Jeol-200CX TEM operating at 200 kV.

3. Results and discussion

The morphology of thermally induced martensite in iron alloys is generally described as lath or plate [12]. Early results suggested that although plate martensites are formed with internal twinning, lath martensites contain no such twins [13]. Later, Patterson and

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Wayman [14] reported the completely twinned structure of the small martensite laths formed in Fe–Ni–C alloys at lower transformation temperatures and showed the influence of alloy composition on the twinning. They also showed the existence of the parallel array of screw dislocations in partially twinned Fe–Ni martensites. Although strain-induced martensite has some basic characteristics of the thermally induced transformation product, such as observed shape change of the transformed austenite volumes, definite habit planes and orientation relationship between two crystal structures, the formation mechanism, morphology and substructure were found to be different [10, 15].

In their detailed study of strain-induced martensite formation in different iron alloys, Tamura *et al.* [1] reported the change of strain-induced martensite morphology with the deformation temperature from thin plate to butterfly. They also noted the existence of a high density of dislocations in strain-induced martensite substructure with the absence of transformation twins and midribs. Later, it was shown that thin strain-induced martensite laths also exhibit twinned substructure, similar to that of thermally induced martensite in Fe–Ni–C alloys and the twins can be observed after careful tilting of the electron microscope specimens [7, 8, 10]. The appearance of the twins was found to be dependent on their orientations with respect to the specimen surface and the observed twins were considered to be transformation twins because the strain-induced martensites are formed with twinned substructures during *in situ* observations [10]. Despite the different formation characteristics of martensitic products there is a common point between these and Tan *et al.*'s [11] results, because both studies have reached the same conclusions, stating the importance of specimen tilting in such observations.

Figure 1a shows transmission electron micrograph of a typical lath-shaped strain-induced martensite formed in the Fe–29.6% Ni alloy during deformation at room temperature which shows typical substructure with a high density of dislocations. The corresponding selected-area diffraction pattern obtained from the austenite-martensite interface is given in Fig. 1b. Indexing of the electron diffraction pattern in conjunction with the bright-field image clearly revealed that the strain-induced martensite habit plane is parallel to $\{\bar{2}25\}_f$ plane of the austenite and there is a Kurdjumov–Sachs type orientation relationship between two crystal structures.

In addition to the needle-like thin strain-induced martensites, larger butterfly martensites were also observed especially during the early stages of the austenite deformation. Fig. 2a shows a transmission electron micrograph of the butterfly-shaped strain-induced martensite. The selected-area electron diffraction pattern obtained from the austenite and butterfly martensite is shown in Fig. 2b. As is seen in the figure, butterfly martensite wings contain a high density of dislocations. Beside this irregular dislocation substructure, it was also observed that there were very fine transformation twins in both wings at their interface. Crystallographic analysis revealed that the

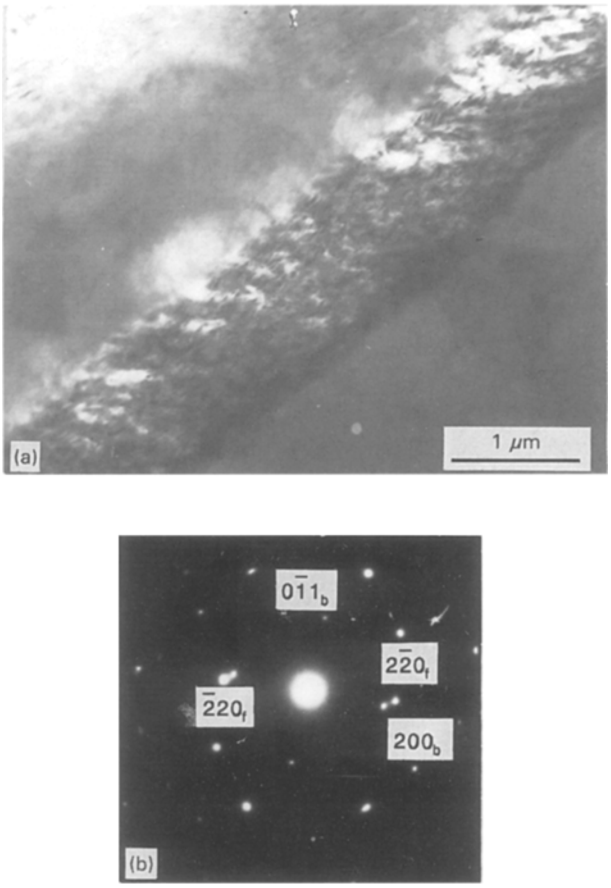
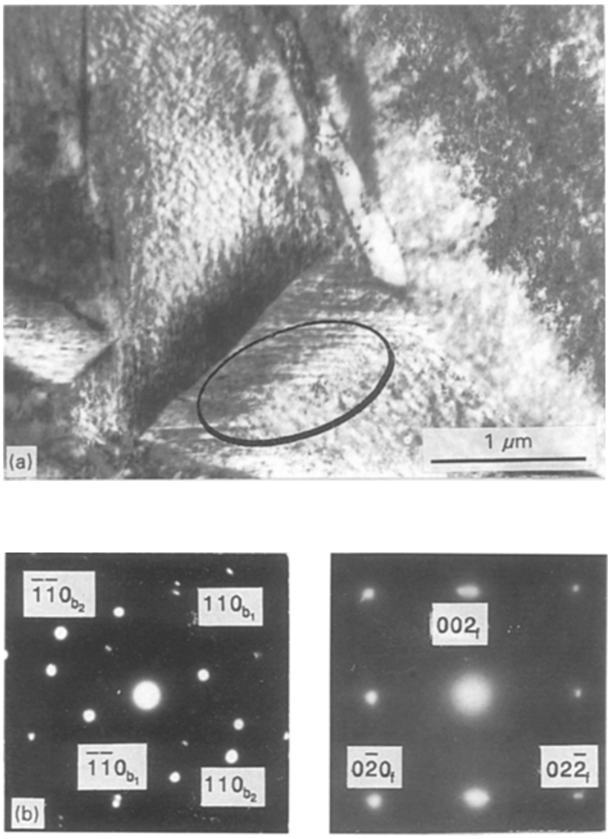


Figure 1 (a) Transmission electron micrograph of a strain-induced martensite showing a high density of dislocations. (b) Indexed selected-area diffraction pattern from the austenite–martensite interface.

transformation twins are formed on the $\{112\}_b$ $\langle 111\rangle_b$ system of the strain-induced martensite and the interface plane of the wings is the $\{111\}_f$ plane of austenite structure. Both wings were found to have the same habit plane as $\{225\}_f$ and a Kurdjumov–Sachs-type orientation relationship with the austenite matrix. Formation of the observed butterfly martensites was explained by Umemoto and Tamura [4] by considering a coupled growth between two wings whose habit planes are related variants of the $\{225\}$. The present crystallographic results obtained for twinned butterfly martensites support this consideration, and the observation of transformation twins at the interfaces of the butterfly wings shows the effect of accommodation distortion which the wings experience during their growth.

The present observations on the substructure of the needle-like thin strain-induced martensites showed that these product structures are also formed with transformation twins. Fig. 3 is a dark-field electron micrograph of an Fe–29.6% Ni alloy strain-induced martensite which is formed at room temperature after 4% deformation of the matrix austenite. A superimposed electron diffraction pattern of the austenite and strain-induced martensite regions is also given in the figure. The minimum necessary deformation level of austenite to strain-induced martensite transformation was determined to be very close to this



Martensite Austenite

Figure 2 (a) Transmission electron micrograph of a butterfly-like strain-induced martensite. (b) Indexed selected-area diffraction patterns taken from the encircled area in (a) and austenite matrix. The twinned martensite diffraction consists of two superimposed patterns (b_1 and b_2).

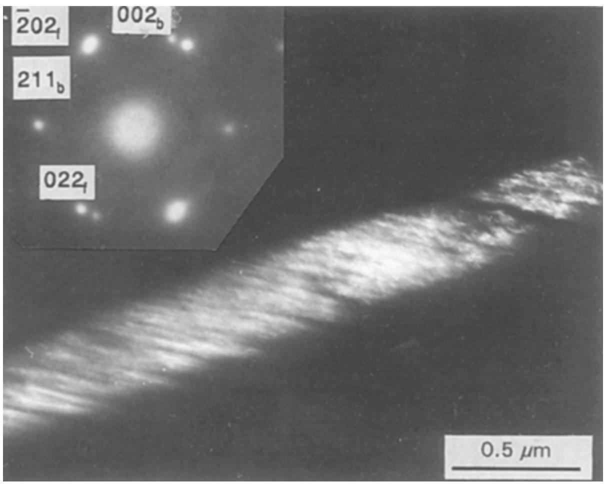


Figure 3 Dark-field transmission electron micrograph of a lath-like strain-induced martensite and corresponding selected-area diffraction pattern.

range, and therefore the observed twins are considered to be transformation twins which are formed due to transformation strains rather than the further deformation of the early formed strain-induced martensites. However, the formation of transformation twins was found to be dependent upon the amount of austenite deformation and no twins were observed in strain-induced martensites formed after relatively large plastic deformations. This indicates

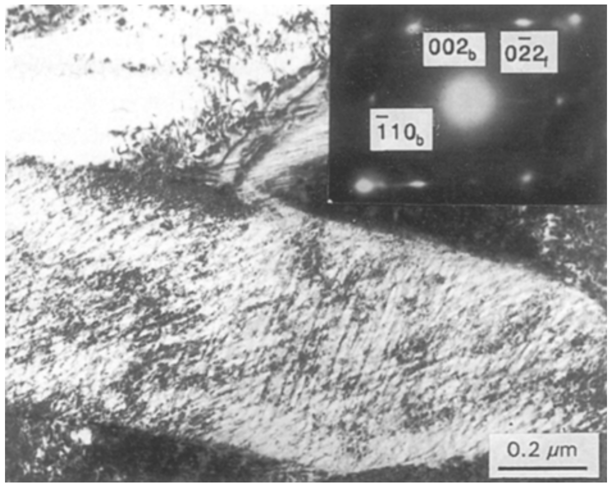


Figure 4 Transmission electron micrograph of a strain-induced martensite with a parallel array of straight dislocations and corresponding selected-area diffraction pattern.

that twinning-type lattice imperfections may be a preferred inhomogeneity during the transformation at smaller deformation ranges.

Figure 4 shows a bright-field transmission electron micrograph of a strain-induced martensite which was formed after 12% deformation of the austenite structure. Despite the lack of transformation twins, a parallel array of straight dislocations was observed inside the product crystal structure. The corresponding electron diffraction pattern is also shown in the same figure. The observed dislocation arrays were found to be parallel to $[1\bar{1}1]$ and $[\bar{1}11]$ directions. Because the only possible slip direction in the b c c-type strain-induced martensite crystal structure is $\langle 111 \rangle$, dislocations were considered to be screw in nature. Strain-induced martensites with a parallel array of dislocations were observed just after their formation and there were no additional plastic deformations following the formation mechanism, and therefore the observed dislocation arrays were formed during the transformation. The existence of parallel straight dislocation arrays was first observed in the untwinned regions of thermally induced plate martensites [14, 16] and it was suggested that a plate martensite may be formed with twinning at the early stages of the transformation, but then grows by a different type of inhomogeneous shear. Patterson and Wayman [14] explained the existence of screw dislocations in the untwinned regions of martensite plates as a result of plastic deformation of the product crystal structure due to constraints produced by the austenite matrix. Although there was no evidence of the transformation twins in the observed strain-induced martensite, the probable source of the parallel dislocation arrays may be the similar constraints during the austenite to strain-induced martensite transformation.

The morphology of strain-induced martensite in several Fe–Ni–C alloys was observed by Maxwell *et al.* [3] and described as parallel "bands" in the austenite matrix. Later observations on the morphology of strain-induced martensite in different iron alloys also showed that in many cases martensites are formed in very close parallel arrays which exhibit relatively large band structures on the macroscopic scale. Those "bands" were resolved with electron microscope observations and it was found that the martensitic regions contain very high densities of dislocations. There was no evidence of the typical transformation twins found in other types of strain-induced martensites [3, 10, 15]. Zhang *et al.* [7, 8] also reported the formation of strain-induced martensite "bands" in an Fe–Ni–C alloy and described the morphology as "packets of parallel laths". According to their results, this type of product structure was found at relatively higher deformation temperatures with the habit plane different from that of butterfly-like martensite. Fig. 5 is a dark-field TEM image of thin strain-induced martensite laths which were induced after 15% plastic deformation. Thin strain-induced martensite laths had a very high density of dislocations as lattice imperfections and careful tilting of the electron microscope specimens revealed the absence of transformation twins in such structures as reported earlier for other iron alloys [1, 7, 8, 10]. The habit plane and the orientation relationship of the observed parallel laths were found to be essentially the same as those for other types of strain-induced martensite morphologies which were examined during the present study. Because only needle-like and butterfly-type martensites were found in the early stages of plastic deformation, it was concluded that the strain-induced bands, which consist of parallel laths, are formed at relatively large deformation ranges.

The relation between the morphology of strain-induced martensite and applied plastic deformation shows that as the deformation is increased at a given temperature, martensites form in more elongated shapes. Although these lath-like strain-induced martensites are parallel to one of the composition planes of butterfly-like martensite wings, they were observed in single configurations. This may indicate that the larger external stresses become more significant than the accommodation stresses and prevent the first plate from inducing transformation to the second one. The plates then form individually by growing parallel to their habit plane on which the external stress resolved in the direction of the shape displacement is greatest. Naturally, formation of the other strain-induced martensites should also be taken into consideration, because their existence may control the formation and the morphology of new martensites.

The observed change in the lath-shaped strain-induced martensite sizes was determined as a function of the plastic deformation by using the formula based on the assumption of martensite plates in thin lenticular shapes [17]

$$\overline{(r/c)} = (3\pi/8)(\bar{F}/\bar{E}) \qquad (1)$$

$$\bar{F} = \overline{(1/c)} \qquad (2)$$

$$\bar{E} = \overline{(1/r)} \qquad (3)$$

where c is the measured maximum thickness of the strain-induced martensites and r is the length. The magnitude of the plastic deformation versus $\overline{(r/c)}$ curve is given in Fig. 6, in which each $\bar{F}$ and $\bar{E}$ value was averaged from ten different measurements. The austenite grain sizes were selected with very close diameters during the experiments to avoid any possible external influence and the butterfly-like strain-induced martensites were not taken into consideration to make a quantitative comparison only for the similar lath-shaped transformation products. As shown in the figure, a considerable change was observed in the mean aspect ratio of the strain-induced martensite laths with the increased amount of plastic deformation at room temperature.

Although the amount of plastic deformation was found to be an important factor influencing the morphology and substructure of strain-induced martensite, it was obtained from the crystallographic analysis of the different strain-induced martensite morphologies during the present work that the habit planes and orientation relationships of all product structures were the same at least up to 20% plastic deformation

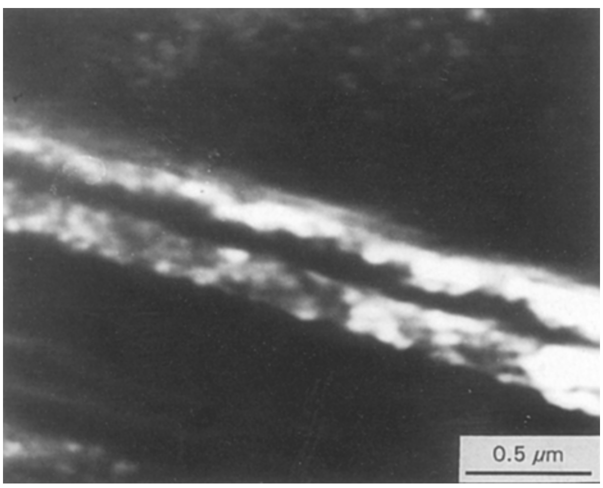


Figure 5 Dark-field transmission electron micrograph of thin strain-induced martensite laths.

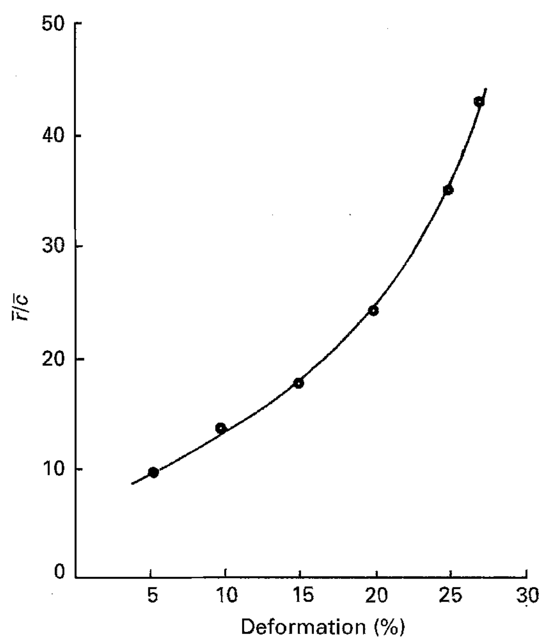


Figure 6 Mean aspect ratio of strain-induced martensites after various amounts of plastic deformation at room temperature.

which was applied to initiate the transformation at room temperature. As was noted earlier in other reports [7, 8, 18], the crystallographic measurements may easily be influenced by large plastic deformations. Therefore, the results reported for the strain-induced martensites formed under these conditions cannot be obtained with reasonable accuracy and the reported difference in the crystallographic parameters of strain-induced martensites formed at large plastic deformations may be caused by the external effects.

4. Conclusions

1. The strain-induced martensite is formed in butterfly- and also lath-like shapes depending upon the amount of plastic deformation which was applied to induce the transformation.

2. Despite the observed differences in the morphology of strain-induced martensites, the habit plane and the orientation relationship were found to be unique as $\{225\}_f$ and Kurdjumov–Sachs, respectively, at least up to a reasonable deformation range which could not destroy the feasible character of the crystallographic analysis.

3. The butterfly- and lath-like strain-induced martensites which were formed just after the minimum amount of plastic deformation necessary to induce transformation, have $\{112\}_b$ $\langle 111\rangle_b$-type transformation twins.

4. The lath-shaped strain-induced martensites formed with no transformation twins at relatively higher deformations, may exhibit a parallel array of straight screw dislocations along $\langle 111\rangle_b$ directions as a transformation inhomogeneity.

5. The strain-induced martensite laths were formed with more elongated shapes at higher plastic deformation ranges.

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