New Observation of Martensitic Morphology and Substructure Using Transmission Electron Microscopy

YU HUA TAN, DE CHANG ZENG, XI CHUN DONG, YUE HUI HE, and SHU QIN HU

The influence on the apparent martensitic morphology and substructure of tilting a thin-foil specimen through various angles in the transmission electron microscope was studied in detail. It was found that the amount of laths with internal twins in low-carbon steel after quenching from high temperature is more than 40 pct; the microstructure of medium- and high-carbon steel after quenching from high temperature is completely twinned martensite. Distinguishing twinned martensite by observation of martensitic morphology and substructure using a thin-foil transmission electron microscope seems rather unreliable. Previous reports that the volume fraction of lath martensite is about 100 pct in low-carbon steel and 75 to 90 pct in medium-carbon steel quenched from high temperature perhaps need reassessment. The conditions under which internal twins within martensite could be observed in the transmission electron microscope are also discussed.

I. INTRODUCTION

SINCE dislocated and twinned martensite were discovered in the early 1960s,[1] electron microscopy has been employed as a means to discern the two major types of martensite in iron-base alloys.[2,3,4] Martensite in which internal twins are seen in the transmission electron microscope was called plate martensite, and martensite comprising a high density of tangled dislocations and essentially no fine twins was termed lath martensite.[2–7] Numerous investigators[4,8–10] have shown that there are only a few twins within lath martensite. Internal twins were not found in the lath martensite of Fe-V, Fe-W, Fe-Sn, Fe-Mn, and Fe-Mo alloys.[10–13] More twins within lath martensite were observed in the 9Ni-0.24C and 9Ni-7Co-0.24C[9] and 5Ni-3.8Cr-0.27C steels.[14]

Speich and Lesite[15] showed the relationship between the volume fraction of lath martensite and carbon contents for Fe-C alloys; the relative volume percentage of lath martensite was near 100 pct for 0.2 pct C steel and 75 to 90 pct for 0.4 to 0.6 pct C steels.

The purpose of this article is to study the effect of tilting thin-foil specimens in the transmission electron microscope on martensitic images and the reliability of identifying twinned martensite by this technique.

II. EXPERIMENTAL PROCEDURE

The chemical compositions of the steels studied are given in Table I. Specimens 10 to 20 mm in diameter and 10-mm thick were quenched from 820 °C, 900 °C, 1000 °C, 1100 °C, 1200 °C, and 1300 °C, respectively. The austenitizing time was 10 minutes in all cases. In steel 20, the cooling medium depends on the austenitizing temperature; when the austenitizing temperature is 1000 °C or above, water was used, whereas below this temperature, brine was used. All other steels were oil quenched from the austenitizing temperature.

The martensitic morphology and substructure of these steels were studied in an S-570 scanning electron microscope, an H-800 transmission electron microscope, as well as in an optical microscope. Thin foils were prepared by grinding and mechanical polishing to 40 μm, followed by electrolytic polishing in a solution consisting of 10 to 20 pct perchloric acid and 80 to 90 pct alcohol at −10 °C in a CE-10 twin-jet polishing unit.

III. RESULTS AND ANALYSIS

A. *Low-Carbon Steels*

The microstructure of low-carbon steel 20 quenched from 980 °C consisted of lath martensite with many intersecting packets. Each packet was composed of dark and light contrast blocks, as shown in Figure 1(a). At 1300 °C, many coarse light-contrast plates emerged in the microstructure, as indicated in Figure 1(b). At higher magnification (Figure 1(c)), these plates appear to be composed of parallel fine platelets with thicknesses ranging from 0.05 to 1.5 μm and 5.5 to 11.4 μm in length.

Figure 2 is the transmission electron microscopy (TEM) micrograph of steel 20, showing many laths possessing internal twins.

It is important to note that changes in the appearance of microstructure within each lath may occur when the specimen is tilted in the transmission electron microscope. The image shown in Figure 2(c) was obtained by tilting the specimen through an angle of 4 deg. Compared with Figure 2(a), two points can be made concerning the image in Figure 2(c). First, twins disappeared completely within a lath located at the center of the photograph; and second, straight interfaces between laths became curved; the characteristic appearance of lath martensite was lost.

Why is it that tilting the specimen could cause such

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Table I. Chemical Compositions of Steels Studied (Wt Pct)

Steel	C	Mn	Si	Cr	Ni
20	0.19	0.70	0.25	—	—
20Cr2Ni4A	0.20	0.42	0.19	1.68	3.70
40Cr	0.43	0.66	0.35	0.95	—
T11	1.12	0.25	0.10	—	—

pronounced changes in the morphology and substructure of martensite? This is because tilting the specimen deviates twin planes from Bragg's condition. The twin planes are not in a proper position for the diffraction of electrons and are unable to reduce the intensity of the transmitted beams. Thus, the image of twins vanishes. Figure 3 is a schematic illustration of this variation. In Figure 3(a), *T* is a local twin within a martensite lath; it produces a strong diffracting effect and causes the intensity of the electron beam to be diminished remarkably, as illustrated in Figures 2(a) and 3(a). A lighter contrast outside the dark rectangle is obtained because there are little diffracting effects; the intensity of transmitted beam is higher (Figure 3(a)). After tilting the specimen through 4 deg, the image of this twin disappears fully in Figure 3(b), because the orientation of the twin is substantially deviated from Bragg's condition and diffraction of electrons does not occur. But the orientation of the other plane [such as plane AB in Figure 3(b)] is close to Braggs's condition and results in a diffraction, and so the diffraction contrast image of plane AB appears in Figure 3(b).

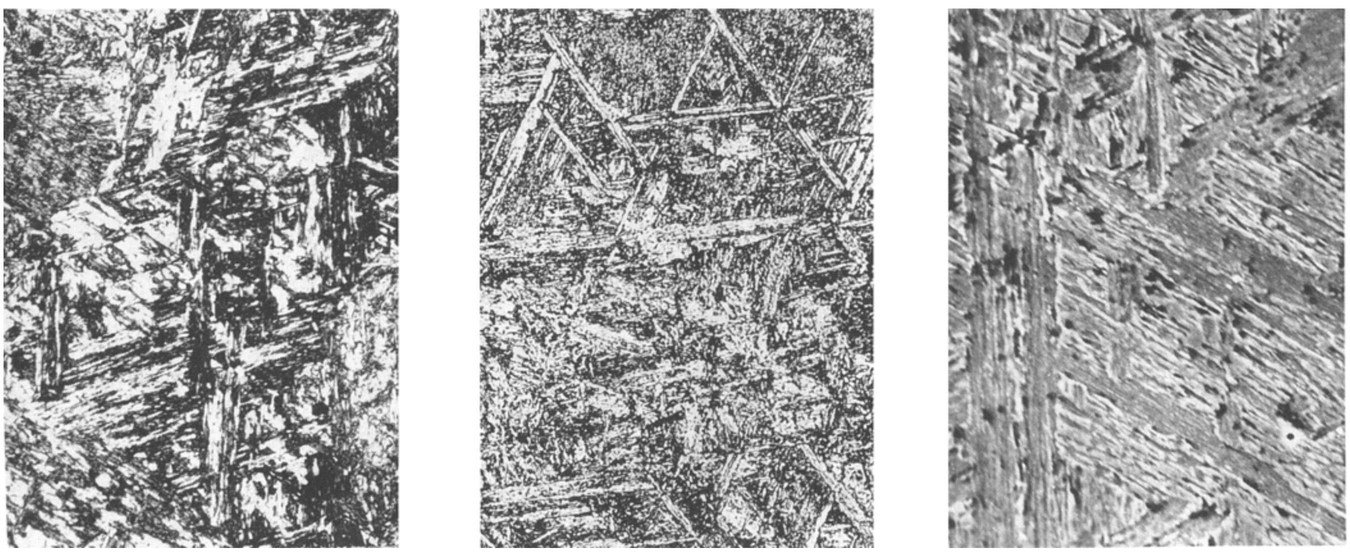
It was found by repeatedly tilting 20 specimens of steel quenched from a high temperature in the transmission electron microscope that the amount of laths, with some internal twins, was 40 pct. This value is much less than that reported by Speich and Lesite,[15] who gave the volume fraction of lath martensite without twins in a 0.2 pct C steel to be about 100 pct.

The transmission electron micrograph of tilted specimens of the $20Cr_2Ni_4A$ steel is shown in Figure 4. Before tilting, the diffraction contrast image of each twin plane is thin, as shown by the dark contrast streaks in Figure 4(a); when tilting the specimen through 2.5 deg, the diffraction contrast image of the twin planes becomes broader due to the increase of the angle included between twin plane and electron beam (Figure 4(b)); after tilting through 7 deg, all of the twins within the martensite vanish (Figure 4(c)). It should be mentioned that the contrast of images in the left and right upper corner of Figure 4(a) also varies with tilting. When tilting the specimen through 2.5 deg, the internal twins in a small plate martensite (0.5×0.21 μm) in the left upper corner vanish; internal twins emerge in a small plate martensite (0.6×0.38 μm) in the right upper corner. In contrast, when tilting through 7 deg, an entirely different variation in the appearance of internal twins and contrast of twin images is produced (Figure 4(c)).

Selected area diffraction patterns before and after tilting the specimen through 7 deg are illustrated in Figures 5(a) and (b), respectively.

B. *Medium-Carbon Steels*

Fine plate martensite was produced on quenching the 40Cr steel from 860 °C; when the austenitizing temperature is raised, the quenched microstructure contains dark and light double contrast martensite, as shown in



(*a*) (*b*) (*c*)

Fig. 1—(*a*) and (*b*) Optical micrographs and (*c*) scanning electron micrograph of steel 20 quenched from 980 °C (Magnification (a) 500 times) and 1300 °C (Magnification (b) 400 times and (c) 2000 times, respectively).

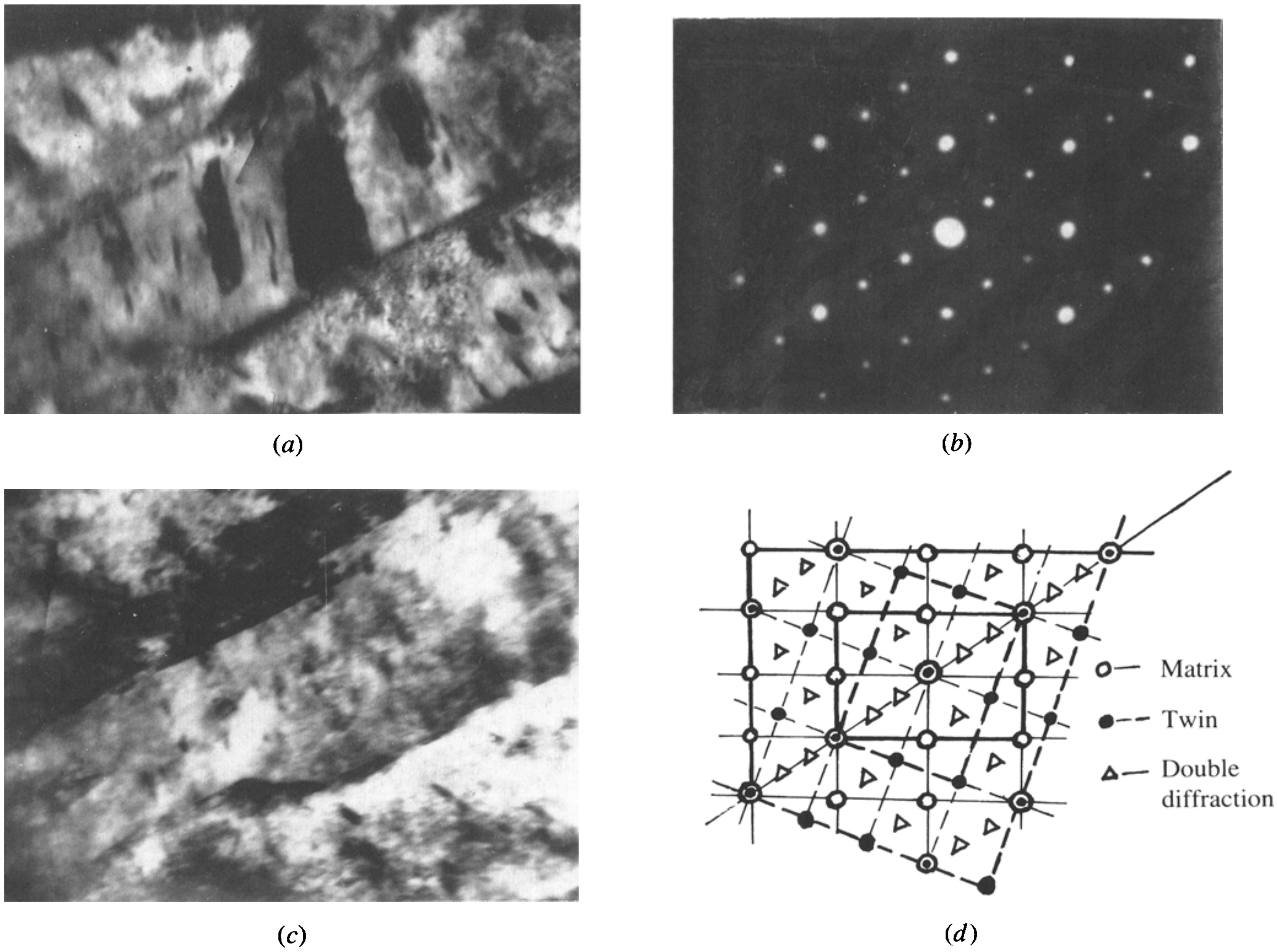


Fig. 2—(*a*) and (*c*) TEM photographs and (*b*) selected area diffraction pattern of twins in steel 20 quenched from 1300 °C. (a) Magnification 40,000 times, (b) specimen not tilted, (c) 4 deg tilted (Magnification 40,000 times), and (*d*) indexed pattern of (b), showing internal twins.

Figure 6(a). Such martensites are present with a fibrous morphology and are built of many fiber clusters, designated by the authors as fiber martensite. At a magnification of 400, platelike characteristic can be observed in the light region of the fiber martensite [Figure 6(b)]. Figures (c) and (d) are micrographs of the light and dark regions of the fiber martensite, respectively. The dimension of these martensite plates is (16 to 28) μm × (0.5 to 3.0) μm × (0.02 to 0.18) μm. Only when the plane of the specimen observed is parallel to the habit

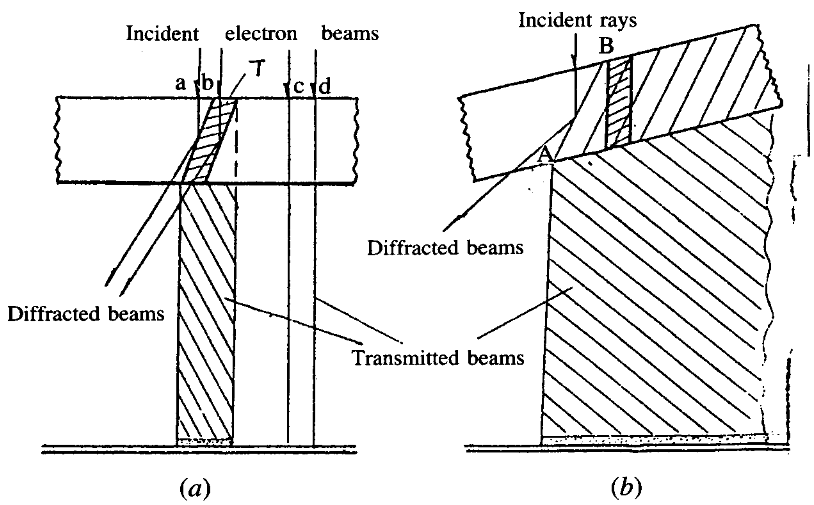
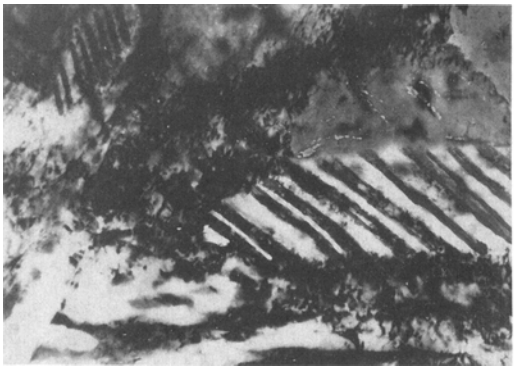
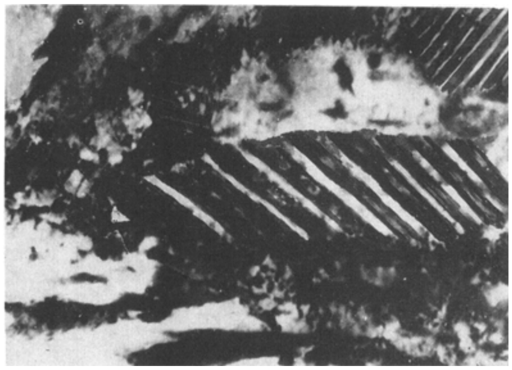


Fig. 3—Schematic diagrams showing the effect of tilting on the variation of diffraction contrast of images (*a*) before tilting and (*b*) after tilting 4 deg.



(*a*)



(*b*)

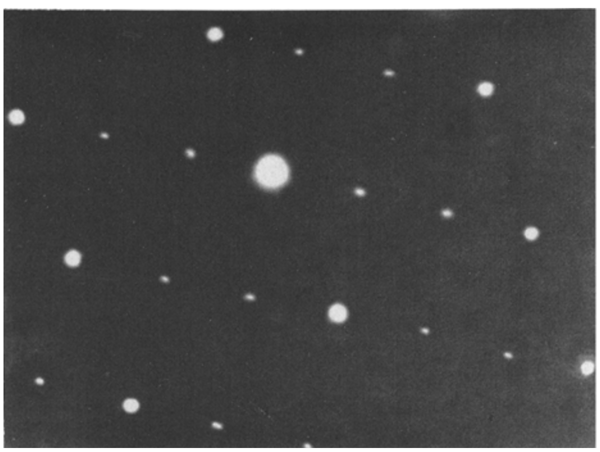
(*c*)

Fig. 4—Transmission electron micrographs of $20Cr_2Ni_4A$ steel after quenching from 900 °C: (*a*) not tilted, (*b*) 2.5 deg tilted, and (*c*) 7 deg tilted (Magnification 40,000 times).

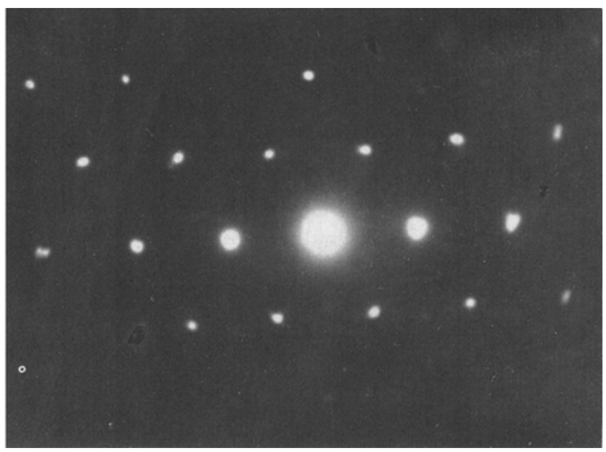
plane of martensite, can micrographs such as that shown in Figure 6(a) be acquired.

The three-dimensional morphology of martensite in Figure 6 is schematically shown in Figure 7. It is comprised of many martensite plates, big and small, essentially parallel to each other and is termed fiber martensite clusters by the authors. Dark and light regions in Figure 6(a) are its horizontal and vertical sections, respectively, and it is easier to etch the former than the latter, as the amount of interface is much greater in the former than in the latter. In addition, differences in crystal orientation will also result in different rates of attack.[20]

Many martensitic plates possess internal twins, as illustrated in Figure 8. The length of the martensite plate with internal twins in Figure 8(a) is about 3.8 μm, and the thickness is about 0.63 μm, which corresponds to the fine plate martensite observed in Figure 6(c). Figure 8(b) shows three martensite plates with a great



(*a*)



(*b*)

Fig. 5—Selected area diffraction patterns for $20Cr_2Ni_4A$ steel quenched from 900 °C: (*a*) not tilted and (*b*) after tilting through 7 deg.

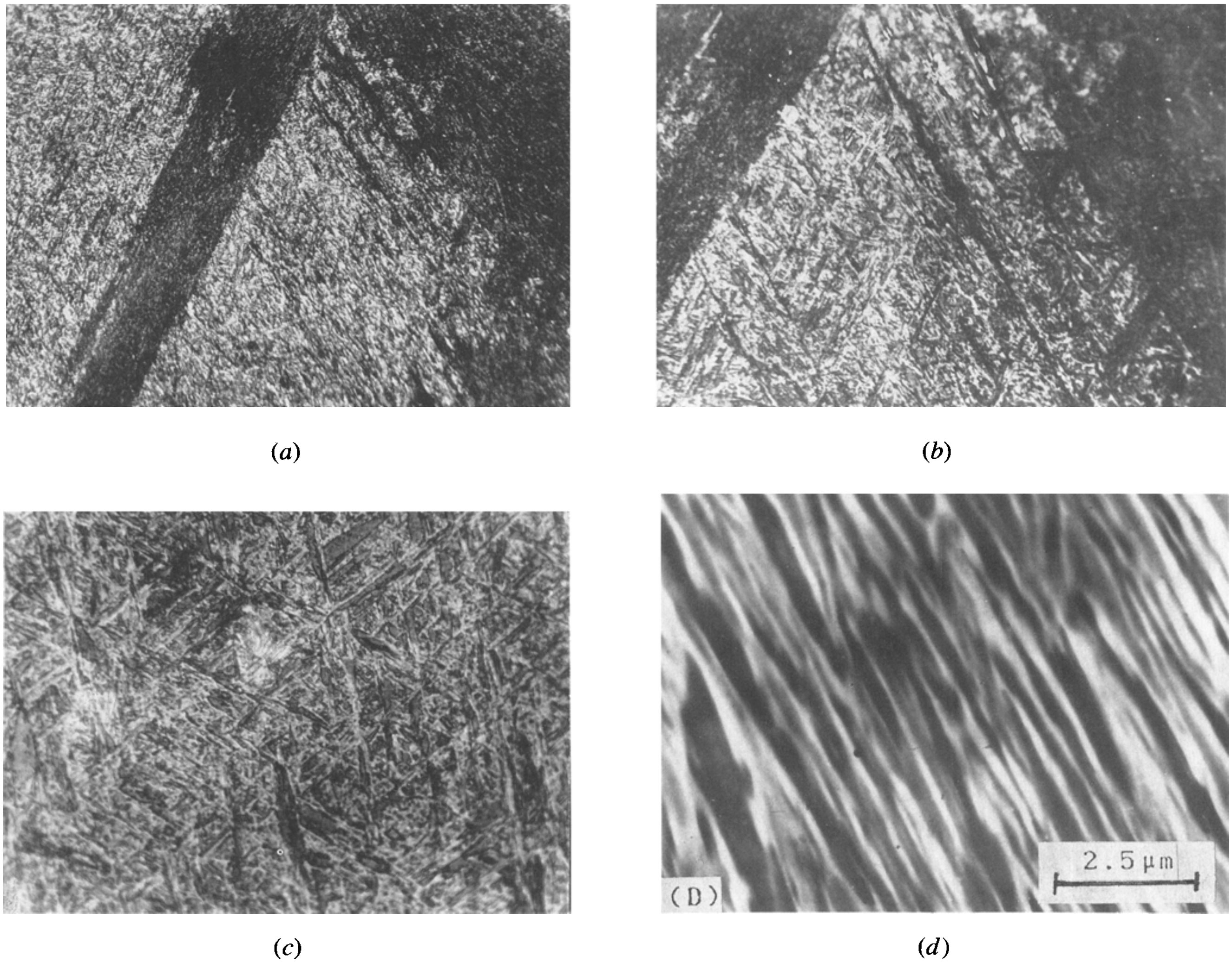


Fig. 6—(*a*) through (*c*) Optical micrographs (Magnifications 250, 400, and 1000 times, respectively) and (*d*) scanning electron micrograph for 40Cr steel quenched from 1150 °C and tempered at 200 °C for 1 h.

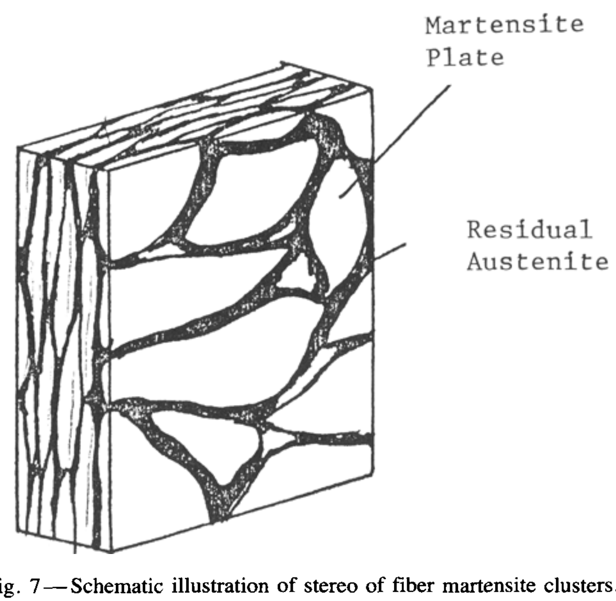


Fig. 7—Schematic illustration of stereo of fiber martensite clusters.

deal of internal twins, parallel to one another; their plate lengths are 0.90 to 1.73 μm and their thicknesses are 0.25 to 0.37 μm, corresponding to the extra-fine plate martensite in Figure 6(c), in which platelike appearance cannot be observed by optical microscopy. Figure 8 cogently shows that fine or extra-fine plate martensite is twinned martensite. All martensite with a plate morphology currently observed under the optical microscope, whether coarse or fine, is twinned martensite.

Speich and Lesite[15] found that in quenched medium-carbon steels (0.4 to 0.6 pct C), plate martensite with internal twins occupied only 10 to 25 pct; the rest was lath martensite without internal twins. This is not in agreement with our observations.

As the microstructure of 40Cr steel quenched from high temperature should be classified as twinned martensite, why is it that only a martensite plate in the center of Figure 8(a) exhibited internal twins while none was observed in the other martensite plate just above it? The absence of image contrast from twins occurs under either one of the following two conditions.

(1) The angle included between the twin plane and electron beam is so large that the diffraction contrast images

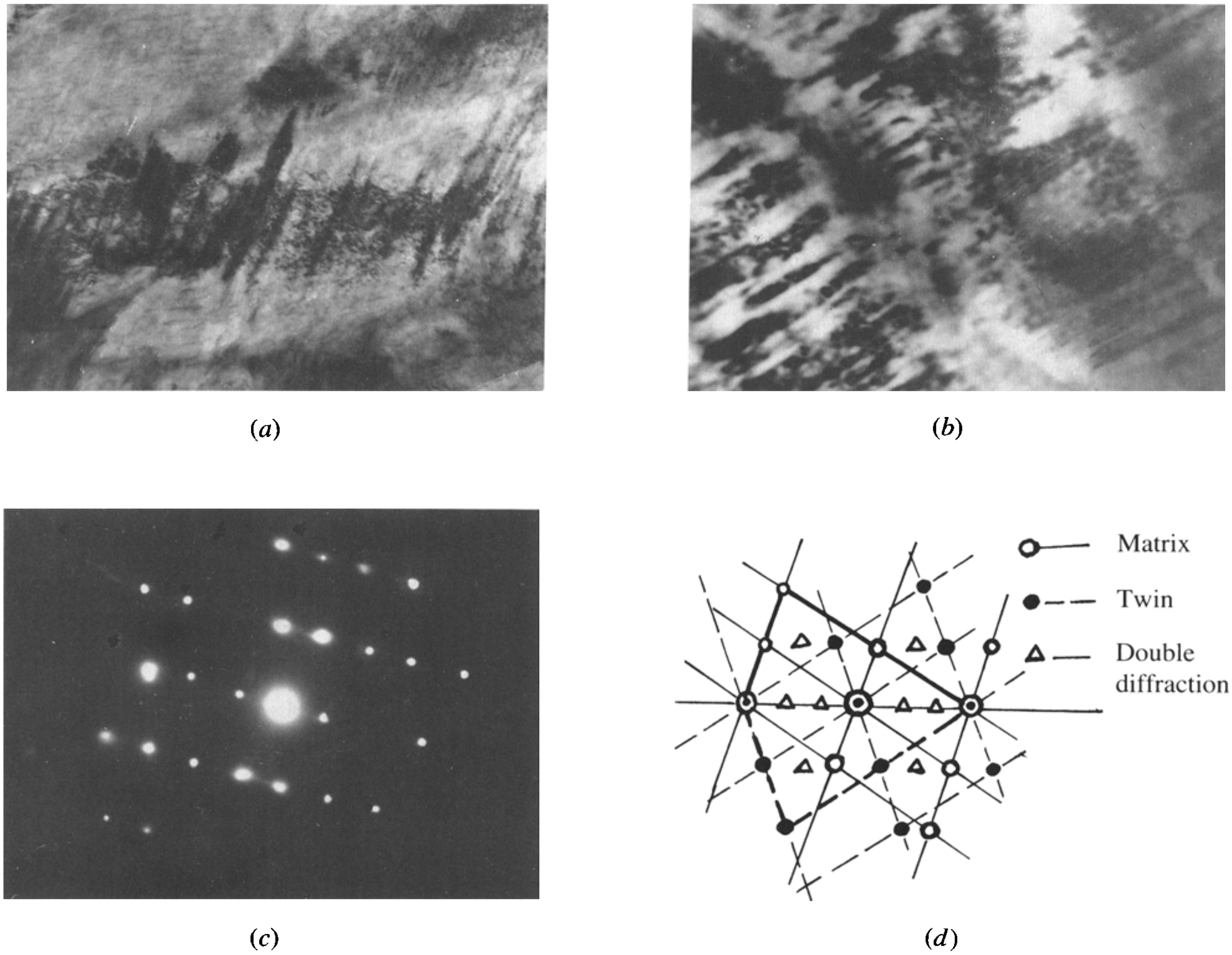


Fig. 8—(*a*) and (*b*) Transmission electron micrographs and (*c*) selected area diffraction pattern of 40Cr steel, 1150 °C oil quenched. (*d*) Indexed pattern of (b), showing internal twins (Magnification 40,000 times).

of neighboring twin planes overlap, as shown in Figure 9(a).
(2) The angle mentioned above is not large, the image of internal twins may emerge when the specimen gets thinner, because the diffraction contrast images of neighboring twin planes do not overlap, as shown in the left of Figure 9(b). If the thickness of specimen after jet thinning is different from place to place, the coincidence of diffraction contrast images of neighboring twin planes may occur, as shown in the right of Figure 9(b).

In the first case, no matter how much the specimen is tilted, internal twins are not imaged, since if the angle tilted is too large, the thickness through which the electron beam passes is enhanced, and consequently, the intensity of the electron beam will be significantly reduced. In the second case, tilting the specimen through a smaller angle in an opposite direction may provide the possibility of showing up internal twins.

C. *High-Carbon Steels*

The microstructure of high-carbon steel quenched from low temperature consists of extra-fine plate martensite which bears more or less random orientation. Platelike martensite was produced with increasing austenitizing temperature. The size of martensitic plates is increased with increased austenitizing temperature. Figure 10 shows the optical and electron micrographs of T11A steel quenched from 1100 °C. It can be seen from the optical micrograph (Figure 10(a)) that, in addition to coarse plate martensite, a few extra-fine plate martensites emerge.

Figure 10(b) is a corresponding transmission electron micrograph, in which "A" is a martensite plate located at the edge of the hole under study. It has numerous distinctive internal twins, since in that region, the specimen is sufficiently thin. The size of this martensite plate is 3 μm in length and about 0.5 μm in thickness; it corresponds to the fine plate martensite shown in the optical micrograph (Figure 10(a)).

In addition, it can be seen from Figure 10(b) that there is also fully twinned martensite in plain-carbon steel. Except having the midrib, its substructure is analogous to that of the thin plate martensite in Fe-Ni alloys.

IV. DISCUSSION

A. *Conditions Revealing Internal Twins*

It has been shown in the analysis given above that it is inappropriate to discriminate between lath and plate

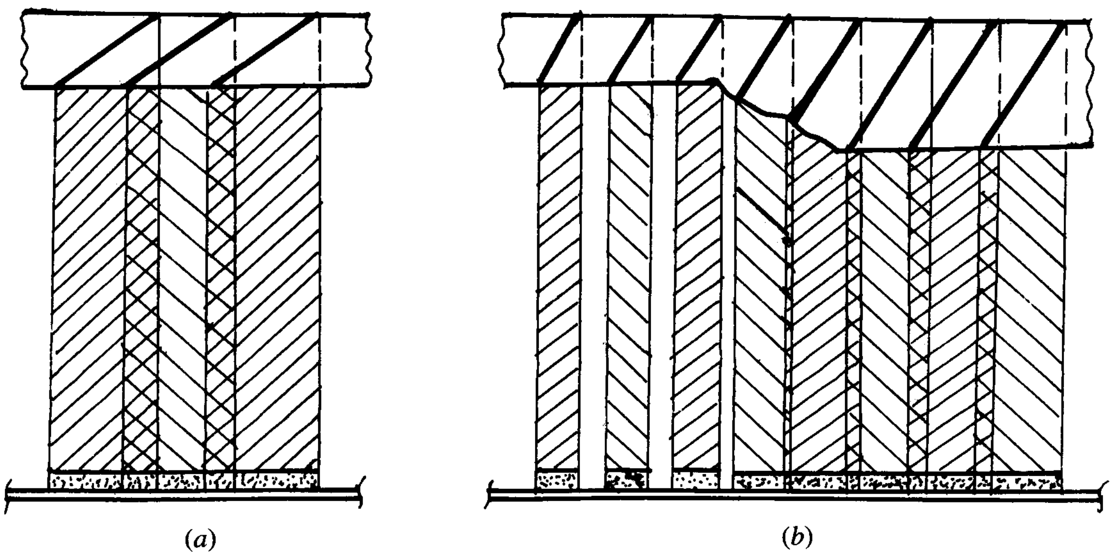


Fig. 9—(*a*) and (*b*) Front elevation of stereo of martensite plate observed under transmission electron microscope.

martensite based on the martensitic substructures observed under the transmission electron microscope. That internal twins cannot be observed within the martensite plates does not mean that the martensite plates had no internal twins. Only under certain conditions is it possible to reveal internal twins by transmission electron microscopy. Figure 11 shows several examples. It is only under the circumstances of Figure 11(a) that internal twins can be imaged in the electron microscope. In the following instances, the images of internal twins may not appear:

(1) when twin planes deviate tremendously from Bragg's condition, internal twins will not be exhibited within a martensite plate; for example, twin planes parallel to the incident electron beam [twin AB in Figure 11(b)] or the angle included between the twin plane and incident ray being much larger than Bragg's angle [twin plane CD in Figure 11(c)];
(2) the distance and orientation of the twin planes are the same as Figure 11(a), yet the diffracted contrast images of the twin planes coincide because the specimen is not thin enough (Figure 11(d)); and
(3) although the orientation of the twin planes is similar to Figure 11(a), the distance between twin planes is small, resulting in overlapping of the diffraction contrast images of the twins (Figure 11(e)).

Figure 11 shows the dependence of the appearance of internal twins on the angle included between the twin plane and electron beam, the distance between twin planes, and the thickness of the thin foil. When the following conditions are satisfied, the image of internal twins will emerge:

(a) $D/\cos\beta < D_K$;
(b) $D \cdot \tan\alpha < 1$; and
(c) $2d \cdot \sin\alpha = \lambda$,

where D_k is a critical thickness (if the distance of travel is greater than this thickness, the intensity of the transmitted beam will be zero); D is the thickness of the thin foil; 1 is the trace distance between twin planes; α is the angle included between the twin plane and electron beam; β is the angle included between the electron beam and the foil normal; d is the spacing of the diffracted atom plane; and λ is the wavelength of the electron beams.

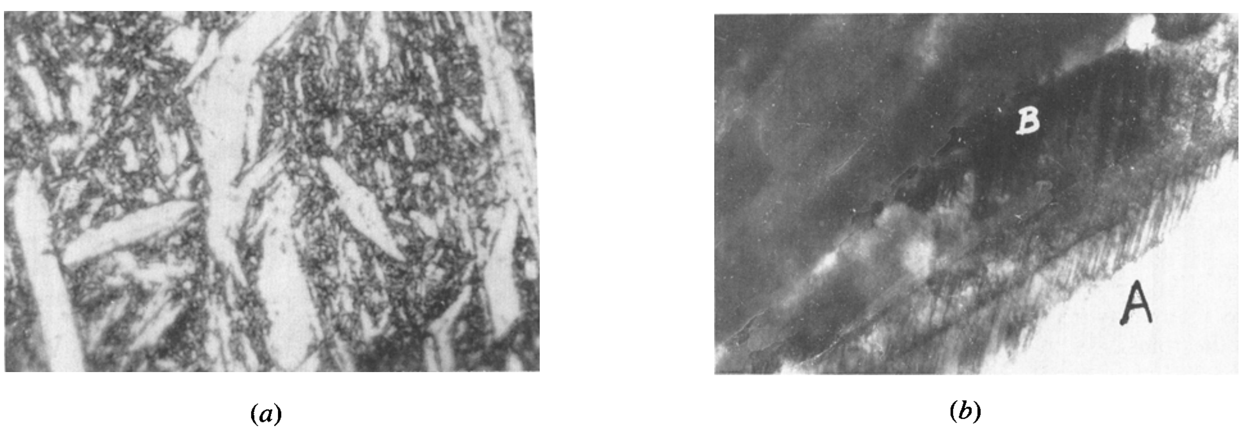


Fig. 10—(*a*) Optical and (*b*) transmission electron micrographs of T11A steel quenched from 1100 °C.

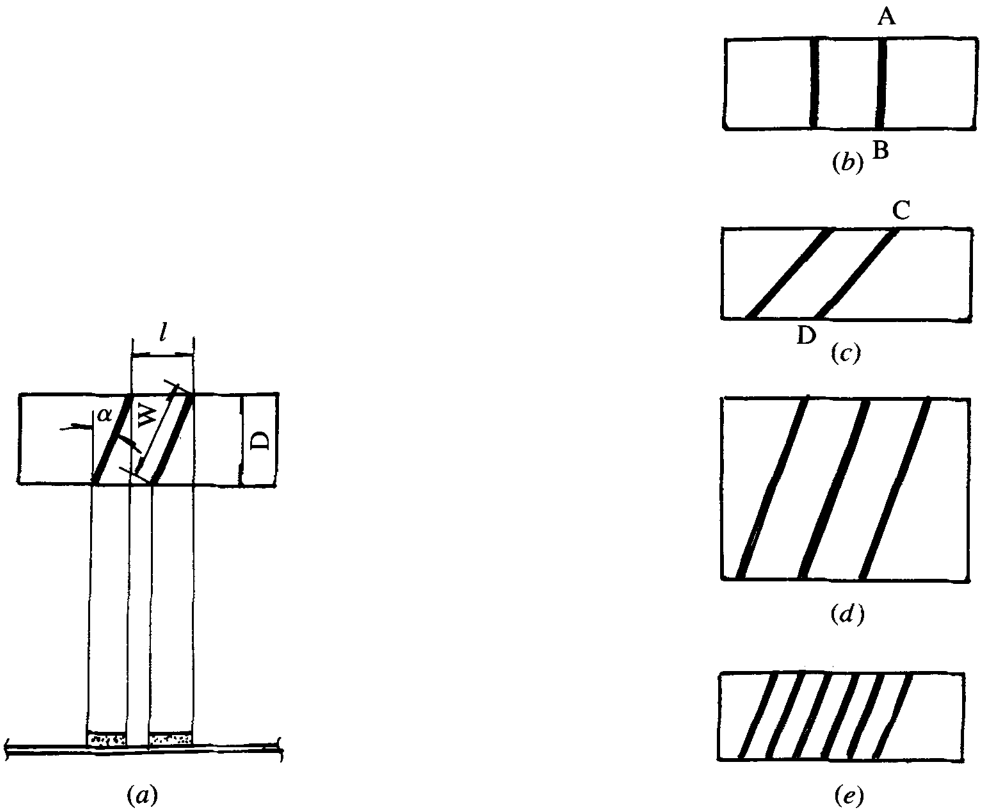


Fig. 11—Schematic illustration for conditions revealing internal twins: (*a*) evidence of internal twins and (*b*) through (*e*) no evidence of internal twins.

When $\beta = 0$ deg, *i.e.*, the electron beam is perpendicular to the specimen, the condition for the emergence of internal twins within martensite will be

(a) $D < D_K$;
(b) $D \cdot \tan \alpha < 1$; and
(c) $2d \cdot \sin \alpha = \lambda$.

(a) and (b) are necessary conditions and (c) is a sufficient condition. In this way, the widespread viewpoint that under the transmission electron microscope the martensite with no internal twins is lath martensite whereas that internal twins are plate martensite needs further clarification, as internal twins which cannot simultaneously fulfill these three conditions are usually invisible. Similarly, carbon content dependence of the relative volume of lath and plate martensite in Fe-C alloys, postulated by Speich and Lesite,[15] may need to be reevaluated.

B. *Proof for Twinned Martensite Obtained in 40Cr Steel after Quenching from High Temperature*

According to the following three observations, it is thought that the fine and extra-fine plate martensite in medium- and high-carbon steels obtained by high-temperature quenching be termed twinned martensite.

(1) No internal twins within martensite plates observed under the transmission electron microscope is not necessarily sufficient proof of the absence of such twins.
(2) The types of substructure within martensite are chiefly governed by the manner adopted during inhomogeneous shear occurring in the martensite transformation. Inhomogeneous shear of martensite will proceed by twinning, and plate martensite will be formed when the formation temperature of martensite is below the starting temperature of twinning deformation.[17,18] It is believed that the starting twinning temperature increases with increased carbon content in steel, as illustrated in

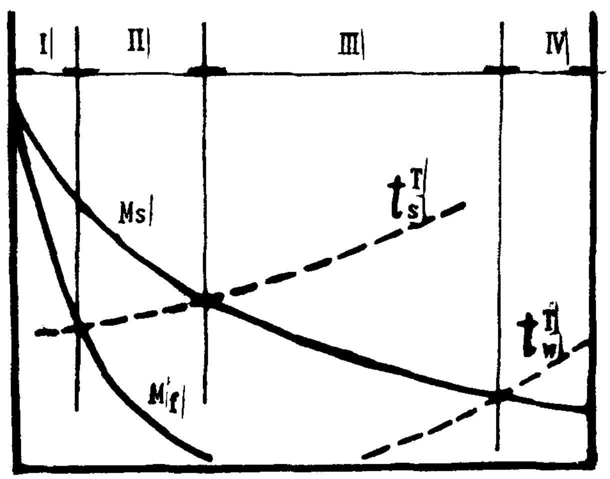


Fig. 12—Schematic diagram showing the change of starting temperature (t_S^T) and M_s temperature with carbon content in steel. t_W^T is the temperature at which inhomogeneous shear of martensite is wholly caused by twinning: I. fully dislocated martensite, II. half-dislocated martensite, III. half-twinned martensite, and IV. fully twinned martensite.

Figure 12. When the carbon content of steel is more than about 0.40 pct, its M_s temperature is lower than the starting twinning temperature t_S^T (approximately 350 °C); hence, inhomogeneous shear in martensite transformation by twinning is complete, and twinned plate martensite is formed instead of dislocated lath martensite. These problems have been investigated in detail in another article,[19] and it has been proposed that martensite should be classified as four types: fully dislocated martensite (such as dislocated lath martensite), half-dislocated martensite (*e.g.*, twinned lath martensite), half-twinned martensite (including twinned plate martensite, butterfly martensite, and lenticular martensite, *etc.*), and fully twinned martensite (for example, thin plate martensite, fully twinned plate martensite).
(3) Considerable internal twins have been observed within martensite in medium- and high-carbon steels [Figures 4(a) and (b), 8(a) and (b), and 10(b)].

V. CONCLUSIONS

The following conclusions can be drawn from this investigation.

1. It has been found from observations of fine microstructures using thin foil transmission electron microscopy that the morphologies of microstructure, sharpness of image of structural details, and extent of evidence of substructures (such as twins and dislocations) will change with the angle of tilting. Structural images exhibited in the transmission electron microscope are changeable and not fixed.
2. Internal twins appearing within martensite under transmission electron microscopy depend on a number of factors, such as the size of martensite plates, the distance and orientation of twin planes, and the thickness of specimens. The lack of observation of internal twins within martensite plates does not necessarily prove that the martensite plates have no internal twins. The currently widespread opinions that martensite with internal twins observed in the transmission electron microscope is plate martensite and that martensite with no twins is lath martensite may not be correct.
3. The conditions revealing internal twins within martensite are: (1) $D/\cos\beta < D_K$; (2) $D \cdot \tan\alpha < 1$; (3) $2d \cdot \sin\alpha = \lambda$. The first two terms are necessary conditions, while the last one is a sufficient condition.
4. On the basis of the above conditions, it is considered that the conclusions and rules concerning the substructure within martensite and the relative volume of lath and plate martensites in Fe-C alloys observed by transmission electron microscopy of repeatedly untilted specimens may not be true.
5. By repeatedly tilting of the specimens, the volume fraction of lath martensite without internal twins was found to be less than 60 pct for low-carbon steels and complete plate martensite with internal twins found in medium- and high-carbon steels.
6. Fully twinned plate martensite was observed in plain-carbon steels.

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