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MULTIPLE TRANSFORMATION TWINNING IN FERROUS MARTENSITES

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ABSTRACT

Transformation twinning in bulk and thin foil transformed specimens of Fe/Ni and Fe/Ni/V/C or Fe/Ni/Mo/C alloys has been studied in detail by high resolution selected area diffraction and dark field analysis which is particularly favorable at high voltages. The microscopy was done on the Berkeley 650 kV electron microscope.

The results show that transformation twinning is complex -- at least two twin systems may operate. The principal twin is a {112} variant which itself may twin on another {112} variant or on a {110} plane to give a small volume fraction of double twins. The double twinning observed in this work is not due to accommodation deformation <u>after</u> transformation.

INTRODUCTION

It is now well known that the transformation substructure of martensite may be dislocated, twinned, or both. Recently we reported[1] the existence of double $(\bar{1}12) - (11\bar{2})$ twinning in lath martensite which is usually non-twinned. It has been argued that this twinning may result from accommodation deformation rather than the transformation shear[2] and it is difficult to unambiguously decide upon the origin of double twinning[3] unless direct observations of the transformation are made and the microstructures compared to those of bulk transformed martensite.

It was decided, therefore, to investigate the twinning in some detail in Fe/Ni alloys and in two other alloys which had previously been investigated with regard to ausforming characteristics. All the alloys have Ms temperatures well below room temperature.

Experimental

The compositions of the alloys used in this study are given in Table 1.

Table 1

Alloy	Ni	C	Mo	V	Fe	~Ms°C
1	33	<0.01			balance	-110
2	35	<0.01			balance	-150
3	24	0.28	4		balance	below -77
4	25	0.3		0.3	balance	

Compositions of alloys after homogenization

Alloys 1 and 2 were made from high purity iron and nickel alloys. They were melted, forged, homogenized and rolled into sheets. The alloys 3, 4 were provided by Ford Motor Company.

The alloys were all transformed as bulk specimens by rapidly quenching from 1100°C into iced brine then liquid nitrogen and in the case of Fe/35Ni finally into liquid helium. Foils were prepared from the transformed sheets and examined at 100 kV and in the 650 kV microscope. The high voltage microscope is superior for the detailed analysis which is required because of the higher resolution, greater penetration and greater accuracy of selected area diffraction at 650kV than at 100kV.

Foils were also prepared of the alloys in the austenitic condition and then transformed either in the cold stage attachment for the Siemens Elmiskop 1 (which unfortunately is non-tiltable) or transformed by dipping into liquid nitrogen and then mounted in the biaxial tilting stage for analysis. In this way the microstructures of martensite formed with and without bulk constraints could be compared. Although the microstructures of the as-transformed foils varied widely, twinned plates were obtained which were identical in substructure to those in bulk specimens.

RESULTS

The results have shown that the twinning substructure is not simple. In fact, the primary twins are themselves twinned (i.e. double twinning).

The proof of this phenomenon requires painstaking exepriments by electron microscopy and selected area diffraction. The analyses involve detailed dark field imaging of all the basic spots in the pattern since the diffraction pattern alone, or the pattern and a single dark field photograph rarely provide unique solutions. It is useful to study orientations where the martensite twins are parallel to the electron beam, e.g. $[113]_\alpha$ foils and also those in which the twins are inclined to the beam direction, e.g. $[122]_\alpha$. The results are shown in figures 1 - 6.

Figure 1(a) shows a partially twinned martensite plate obtained from the Fe-33Ni alloy that was transformed in bulk. The orientation of the martensite phase is $[22\bar{1}]$. Figure 1(c-f) shows a set of dark field images, corresponding to the spots in fig. 1(b), indexed in fig. 2. If the $[22\bar{1}]$ martensite is singly twinned on the $(11\bar{2})_\alpha$ the $[00\bar{1}]$ twin orientation is produced and the pattern would be superposed $[00\bar{1}]$ and $[22\bar{1}]$. If this were true, then dark field images of the spots at the 110 and $\bar{1}10$ positions, i.e. spots C and D, would reveal twin contrast for both of these. However, as the dark field images of fig. 1c, d show, the twin reversal only occurs for one particular 110 position, (i.e. for spot C). The explanation is that the primary twins on $(11\bar{2})$ are themselves twinned on $(\bar{1}12)$ so that the $[00\bar{1}]$ twin of $[22\bar{1}]$ now transforms locally into the $[2\bar{2}\bar{1}]$ orientation. As a result there is no secondary twin reflection at D and hence no reversal of twins in fig. 1(d), and similar is the case in fig. 1(f). However, the extent of the double twinning may be limited, and in this case one expects reversal of contrast. Similar results were obtained for specimens transformed as foils, e.g. fig. 3, 4. The secondary twins are resolved better in fig. 4. The contrast at S in fig. 4 is consistent with secondary twins on $(11\bar{2})$ in the primary twins. The dark field image of the spot marked T" (corresponding to spot C in Fig. 2) reverses the contrast of both primary and secondary twins. This spot is actually the superposition of both (110) primary twin and $(\bar{1}\bar{1}0)$ secondary twin reflections.

These conclusions regarding twinned twins are further confirmed by analysis of the edge-on twins in the fully twinned $[113]_\alpha$ martensite plate of fig. 5. Here the primary twin plane is $(21\bar{1})_\alpha$ which transforms the [113] matrix to $[\bar{1}\bar{1}\bar{3}]$ after twinning, as verified by the diffraction pattern and dark field analysis. However, this example is even more complex. Faint streaks are resolvable on the negative of fig. 5(b) parallel to the <110> directions indicated on this pattern. These streaks cannot be explained by double {112} twinning as in figs. 1-4, but can be explained by secondary {110} twinning both in the matrix and in the primary twin. The $[\bar{1}10]$ twin streak is, of course, the twin of the $[\bar{1}10]$ matrix streak, i.e. these streaks could represent the same transformation twin in the matrix and its primary twin. However, since the diffraction streaks are so weak it is not possible to resolve secondary twins in the image, although magnified photographs of the structure show indications of substructural details, but which cannot be identified. It is interesting to note that the interface in fig. 5 is parallel to $(\bar{7}\bar{3}9)_\alpha$. Assuming a Bain correspondence this plane would be parallel to the $(\bar{5}29)$ of austenite. In most of the cases examined it was found that when the secondary twins were on {112} the martensite plate was partially twinned probably near $\{225\}_\gamma$ habit. On the other hand the fully twinned plates, with a possible $\{259\}_\gamma$ habit showed {110} secondary twins. The results for the partially twinned plates in figs. 1-4 cannot be explained on the basis of {110} twinning and these plates are probably $(225)_\gamma$ type martensites. Although this is a speculative correlation at this stage, we might suggest that {110} secondary twinning may be a characteristic of low temperature $(259)_\gamma$ habits.

It may be mentioned that Oka and Wayman[5] also observed {101} transformation twins in the martensite of an Fe-1.8°C alloy, with $\{259\}_\gamma$ habit. The complexity of the situation is emphasized by the fact that figs. 1 and 5 were obtained on the same specimen and within 10 microns of each other.

It is noted that the twins are often displaced or truncated, and always parallel to $\{011\}_\alpha$ traces, consistent with slip (or possibly twin) deformation (e.g. fig. 3). Dislocations can be resolved between the twins in some cases, but in general the contrast requirements are different for revealing twins and dislocations separately.

There is surface structure resolvable on the broad faces of the twins as can be seen in figs. 1, 3, 4 and some of the complex fringe patterns observed in the dark field images fig. 1c-f may be related to the secondary twinning or to variations in twin thickness, e.g. at ledges. These fringes may also be associated with regular arrays of twin/twin or twin/matrix interface dislocations, but it has been difficult to obtain definitive images even after using several different reflections fig. 1c-f. The fact that these fringes do not change orientation (direction) when different reflections are used rules out the possibility of moiré contrast. Other separate experiments show that complex dislocation arrangements are present in addition to twinning. Figure 6 is an example. The dislocations shown here cannot be a/6<111> twinning dislocations since all such dislocations would be invisible in the $g = [110]$ reflection ($g.b = 1/3$ or 0).

DISCUSSION

These results indicate the possibility of the following shear systems; single {112}<111> twins in α, which themselves may twin on {112}<111> of a different variant or {110}<UVW> twins within primary twins and matrix, and also {110}<111> slip shears. It has been suggested but not proved that $\{225\}_\gamma$ martensites suffer slip and twin shears. Recently, Acton and Bevis,[6] and Ross and Crocker[7] have developed generalized theories of the crystallography of the martensite transformation which involve supplementary shears but not of the type observed here. In fact, there has been little experimental evidence to support that multiple shears operate in the martensitic transformations in steels. An attempt should be made to reconsider the phenomenological theory in terms of twinned twins and slip, and this approach may help to solve the mystery of the $\{225\text{-}259\}_\gamma$ habits for which the type and amount of twinned twins may be an important factor.

It may be argued that the double twinning may result from plastic deformation after transformation. However, the fact that double twins are observed in martensites formed directly in thin foils lends good support for the belief that double twinning is a feature of the transformation itself.

Double twinning has also been detected in lath martensite.[1,8] The absence of reports of double twinning in previous electron microscopic studies of martensites, particularly in view of extensive work done on twinned Fe/Ni alloys,[9,10,11,12] serves to emphasize the necessity of carrying out complete dark field and diffraction analyses to characterize microstructure. However, such analyses are not so feasible when dealing with the usual complex martensites in fully transformed commercial steels.

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References

1. S. K. Das and G. Thomas: Met. Trans., 1970, 1, 325-327.
2. C. M. Wayman: Met. Trans., 1970, i, 2009.
3. S. K. Das and G. Thomas: Met. Trans., 1970, i, 2009 (Responses to 2).
4. O. Johari and G. Thomas: Trans. ASM, 1965, 58, 563-578.
5. M. Oka and C. M. Wayman: Trans. AIME, 1968, 242, 337-338.
6. A. F. Acton and M. Bevis: Mater. Sci. Eng., 1969, 5, 19-29.
7. N. D. H. Ross and A. G. Crocker: Acta Met., 1970, 18, 405-418.
8. D. Huang and G. Thomas: Submitted to Met. Trans.
9. H. Warlimont: Electron Microscopy, 1962 (Acad. Press, N.Y.) pHH6.
10. K. Shimizu: J. Phys. Soc. Japan, 1962, 17, 508-519.
11. G. Krauss and W. Pitsch: Trans. AIME, 1965, 233, 919-926.
12. R. L. Patterson and C. M. Wayman: Acta Met., 1966, 14, 347-369.

Figure Captions

Fig. 1. Martensite in Fe-33Ni transformed in bulk.

A) Bright field image

B) Selected area diffraction of central part of A.

C) Dark field image of spot C $(110)_{t_1} + (\bar{1}\bar{1}0)_{t_2}$. The twins show wedge fringe contrast at W.

D) Dark field image of spot D $(1\bar{1}0)_{\text{matrix}} + (\bar{1}10)_{t_1}$

E) Dark field image of spot E which is $(200)_{t_1}$, or doubly diffracted matrix and t_2 spots.

F) Dark field image of spot F $(\bar{2}\bar{4}0)_{t_1} + (024)_{\text{matrix}}$.

In C the strong fringe contrast is due to wedge thickness changes. The other fringes are parallel to the trace of $(\bar{1}12)$ in the $(00\bar{1})_{t_1}$, and could represent the double twin interface. 650kV

Fig. 2. Calculated diffraction patterns obtained from a martensite in $[22\bar{1}]$ orientation.

a) the pattern if the $[22\bar{1}]_{\text{matrix}}$ is singly twinned on $(11\bar{2})$; superposed $[22\bar{1}]_{\text{matrix}} + (00\bar{1})_{t_1}$.

b) the pattern containing spots due to primary twins on $(11\bar{2})$ and secondary twins on $(\bar{1}12)$; superposed $[00\bar{1}]_{t_1} + [2\bar{2}\bar{1}]_{t_2}$.

c) the pattern if the $[22\bar{1}]_{\text{matrix}}$ twins to $[00\bar{1}]$ and the $[00\bar{1}]$ partially twins to $[2\bar{2}\bar{1}]$. This pattern explains Fig. 1(b).

Fig. 3. Fe/25Ni/0.3V/0.3C thin foil transformed in liquid nitrogen showing partial twinning. The primary twins and fringe contrast are reversed in this dark field image of the 110 twin spot. The twins are truncated parallel to (110) matrix. The broad faces of the twins contain structural details which may be ledges, secondary twins, or interface dislocations (see also fig. 1)

orientation near <3,1,11>. 650kV

Fig. 4. Martensite in Fe/25Ni/0.3V/0.3C, thin foil transformed in the cold stage at -80°C.

A) Bright field image

B) Selected area diffraction

C) Dark field of spot T" in (b). This spot corresponds to the position 'C' in Fig. 2(c).

Notice twin fragments (arrowed) and secondary twin contrast at S (consistent with secondary twin trace). 100kV

Fig. 5. Martensite in Fe-33Ni transformed in bulk.

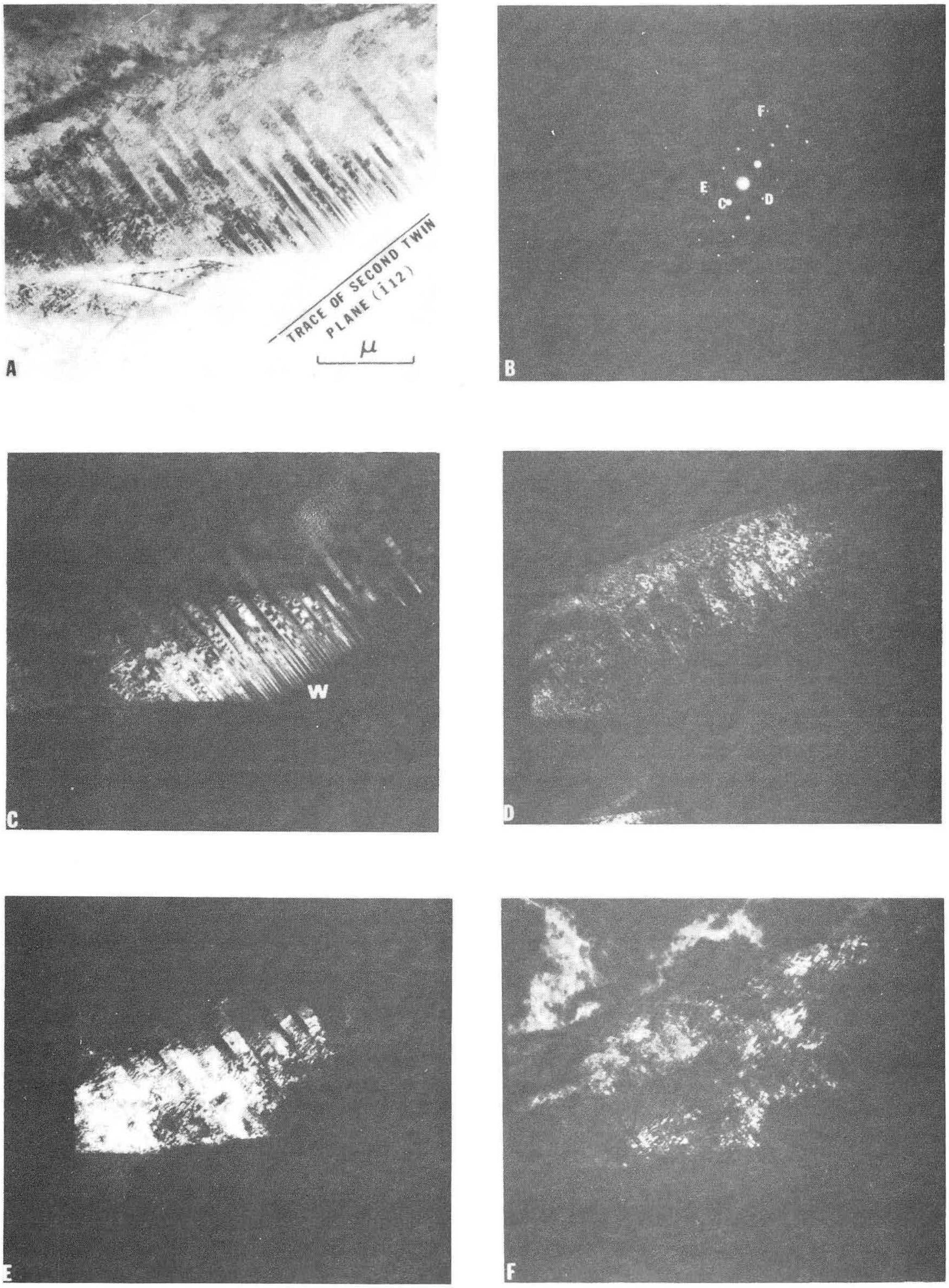
A) Bright field

B) Selected area diffraction pattern showing strong streaks due to $(\bar{1}\bar{2}1)$ primary twins; double diffraction spots (white arrows) and very faint streaks along $[\bar{1}10]$ matrix and $[\bar{1}10]_{twin}$. The $[\bar{1}\bar{1}\bar{3}]$ primary twin spot pattern is superposed on $[113]_{matrix}$.

C) Dark field image of spot C $(\bar{1}10)_{t_1}$ (which also includes the faint streaks). 650kV

D) Dark field image of spot D $(1\bar{1}0)_{matrix}$.

Fig. 6. Dark field image of $(110)_{matrix}$ spot obtained from Fe-33Ni, transformed in bulk showing twin fringe contrast as well as dislocations in two adjacent martensite plates. 650kV



XBB7010-4373

Fig. 1.

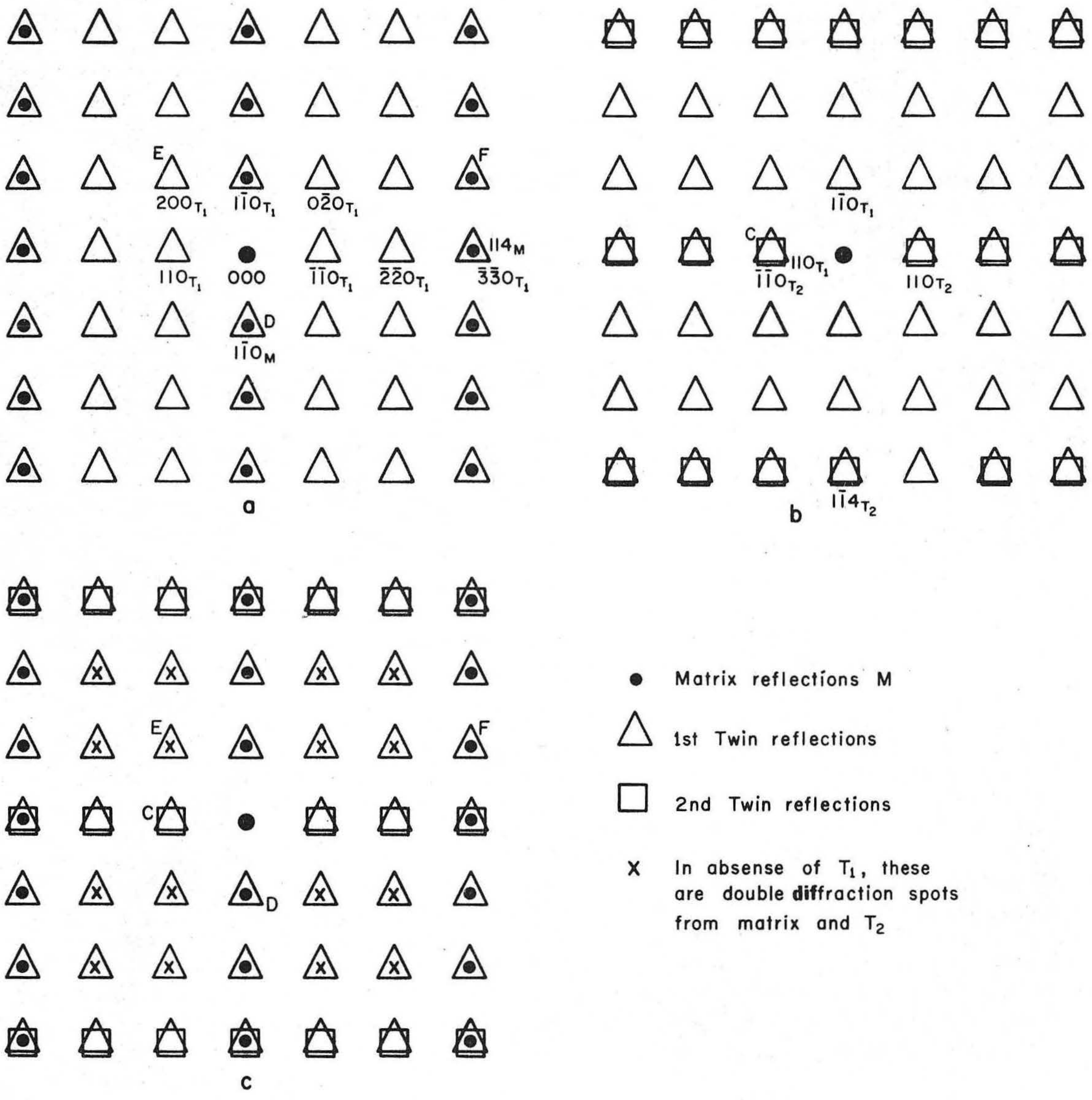


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Fig. 2.

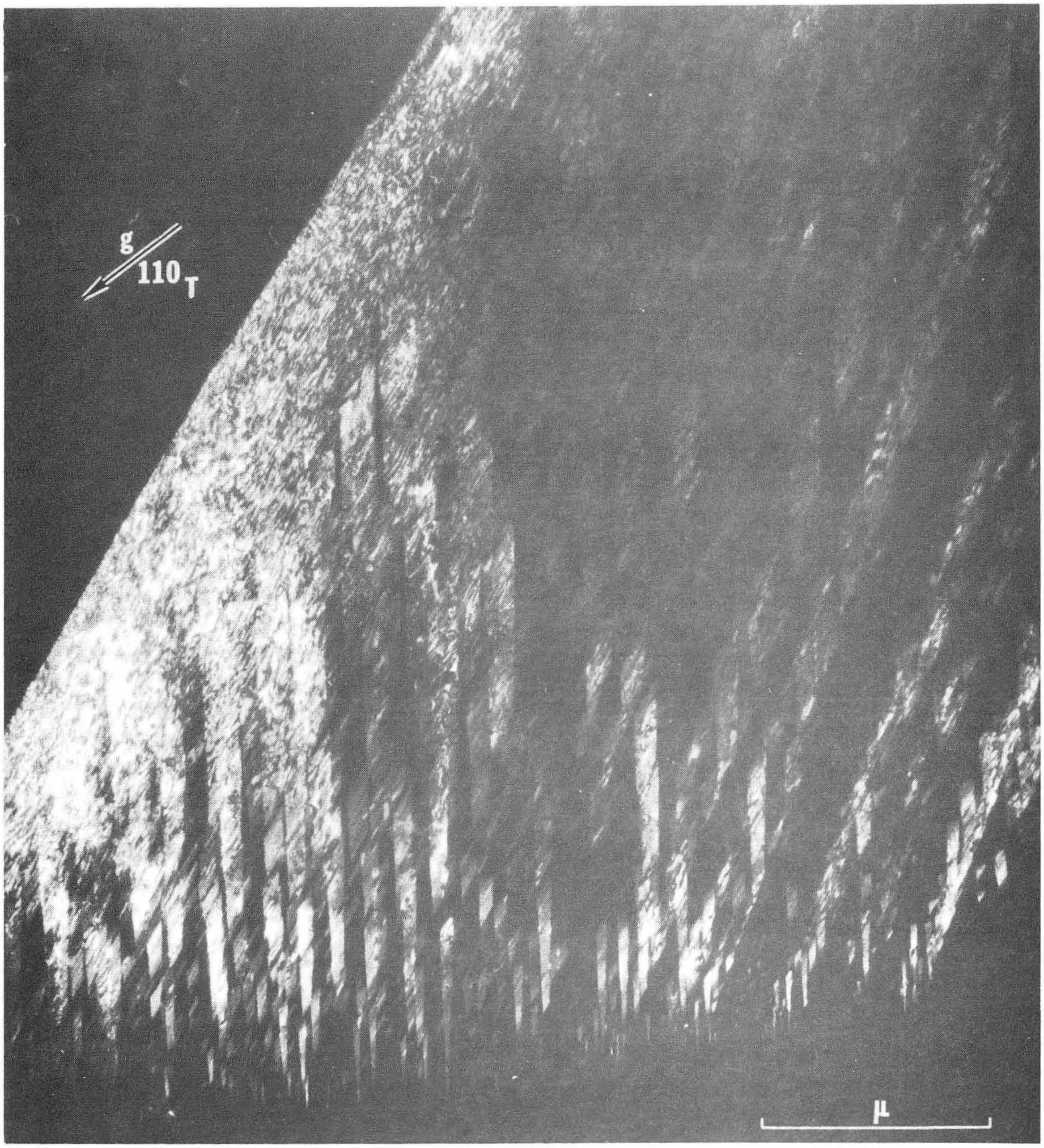

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Fig. 3.

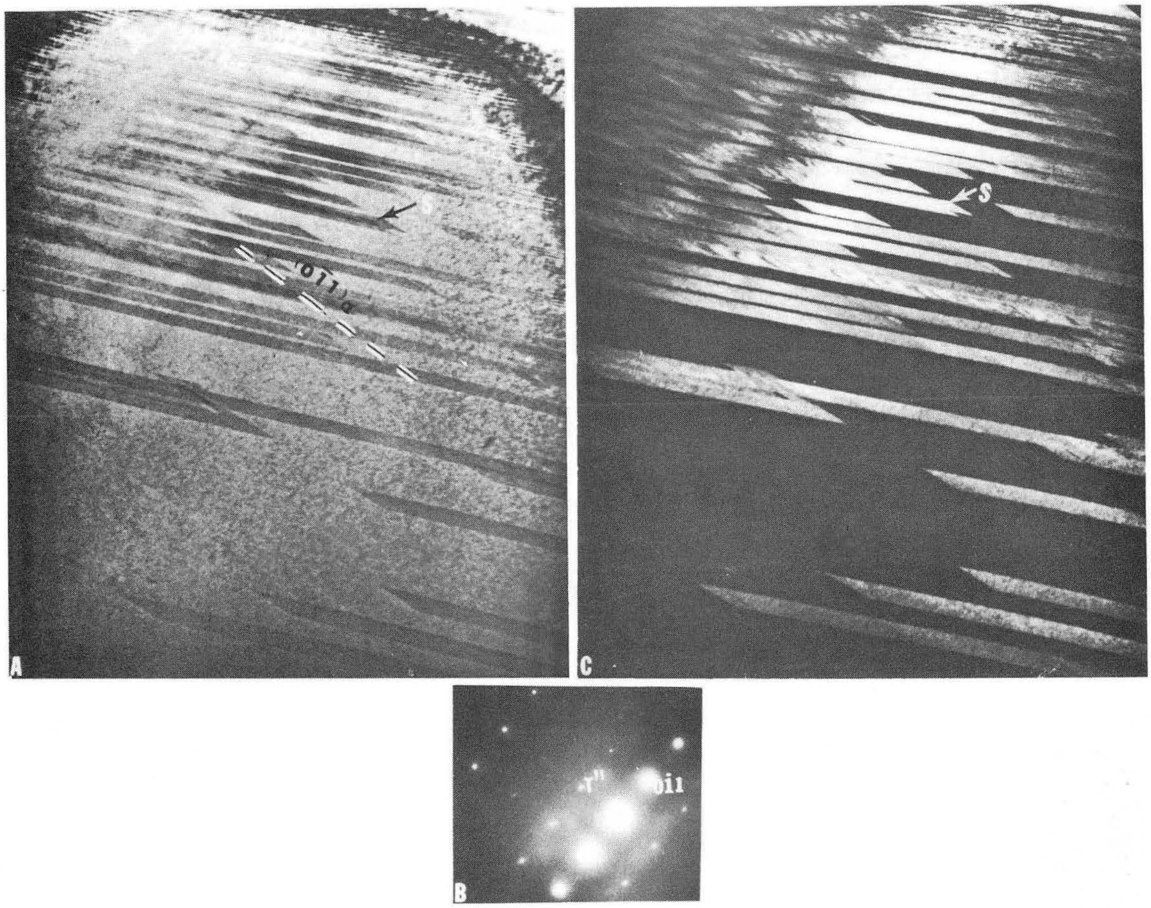

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Fig. 4.

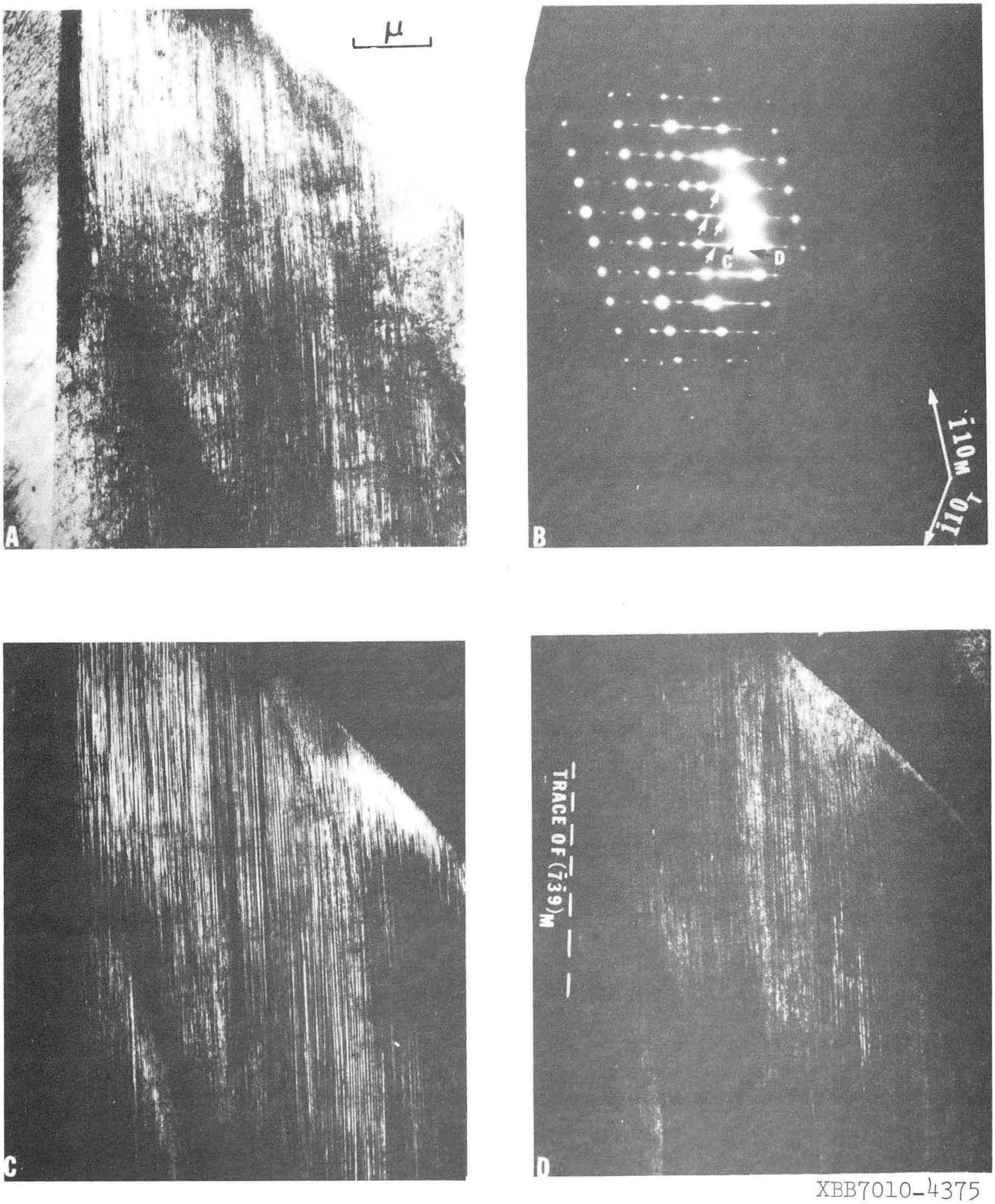

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Fig. 5.

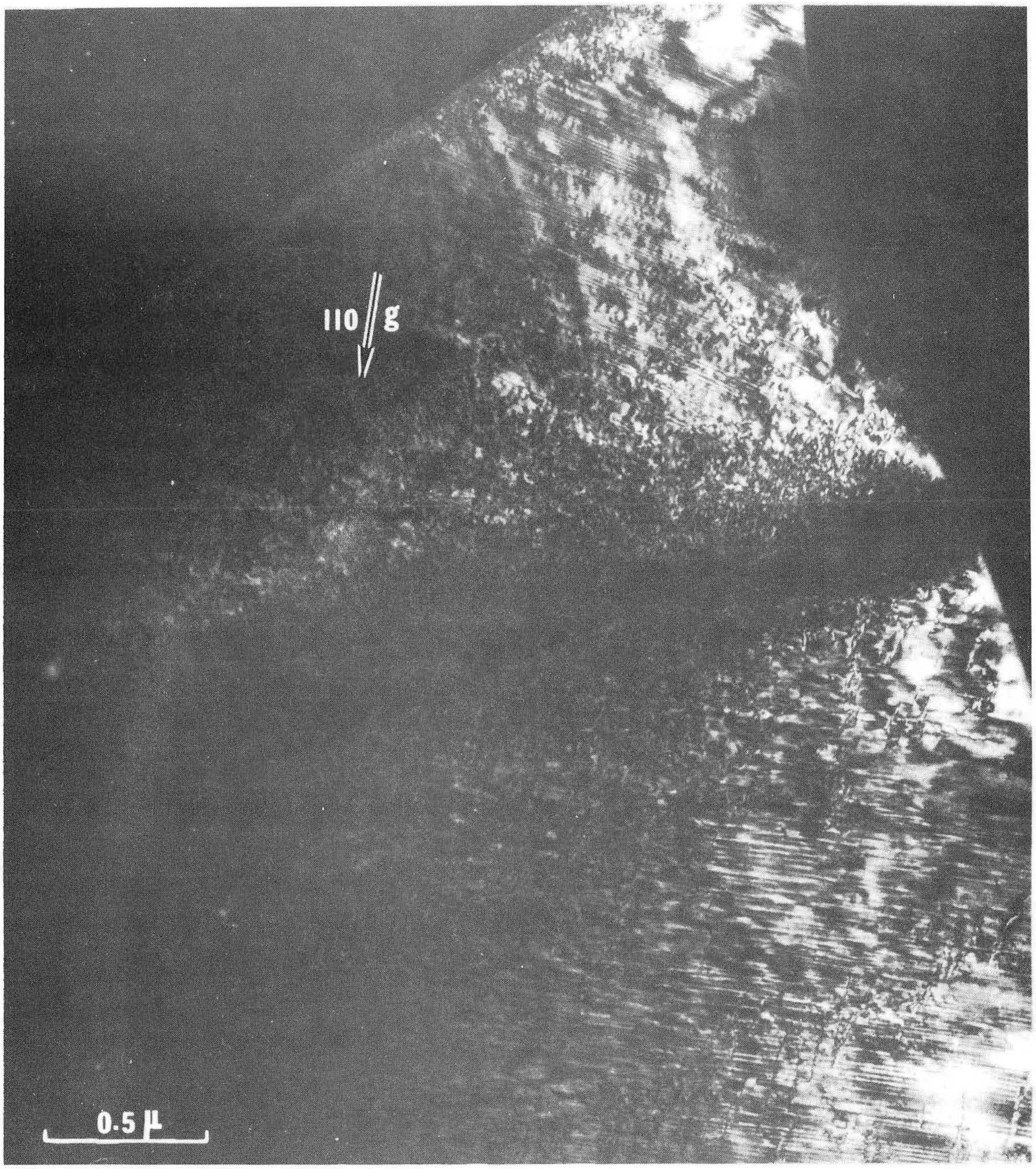

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Fig. 6.

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