Stress Induced Transformations in Beta III Ti Alloy Single Crystals

J. A. ROBERSON, S. FUJISHIRO, V. S. ARUNACHALAM, AND C. M. SARGENT

A combination of X-ray metallographic and transmission electron microscopy techniques have been used to investigate deformation modes and deformation products in Beta III Ti alloy single crystals. Crystallographic slip was observed on {110}, {112} and {113} planes. Twinning occurred on {112}, {332} and {2, 4.8, 4.8} planes. A martensitic transformation of the type proposed by Blackburn and Feeney[1] took place; there was also twinning in this martensitic product.

MECHANICAL twinning has been observed to play an important role in the deformation of beta stabilized Ti alloys.[1-2] This behavior has also been reported in the commercial alloy Beta III, which has the nominal composition (Ti-11.5 pct Mo-5.5 pct Zr-4.5 pct Sn). This characteristic is most clearly evident when the alloy is deformed in the solution treated and quenched condition.[1,3,4] Rack *et al*[4] report that deformation by cold rolling produced {112} twins, whereas Blackburn and Feeney[1] found a less common twinning system, {332} ⟨113⟩, to be operative. These systems were determined by single surface trace analyses. Roberson and Adair[3] in a recent communication have reported that twinning influences the subsequent behavior of Beta III titanium alloy.

In X-ray/metallographic trace analysis, one has to rely on the crystallographic orientation of single crystals which has been determined prior to deformation. Thus, there is always some inaccuracy in the reference direction of the crystals because preceding and concurrent slip inevitably causes rotation of the crystals adjacent to the twins. Because traces are sometimes associated with the products of strain-induced and/or martensitic transformation, it is necessary to reveal the crystallographic structure of such products by a diffraction method. However, X-ray diffraction is not suitable for this purpose because the deformation products are usually small in size and heavily distorted. Thus, transmission electron microscopy (TEM), coupled with electron diffraction has been employed to supplement and to support the optical trace analysis.

EXPERIMENTAL PROCEDURE

Single crystal or coarse grained samples of Beta III titanium alloy were prepared by annealing pre-machined tensile specimens in a purified dynamic atmosphere of helium at 1400°C for 24 h. The specimens were then quenched in water, electropolished, and etched to reveal grain boundaries. In many cases the grain boundaries were normal to the tensile axis and extended completely across the gage section of the tensile specimen. Some of the specimens were tested in tension in this condition, and some of them were sectioned transversely to produce single crystal compression specimens. The orientations of the crystals were determined by Laue back reflection X-ray diffraction prior to testing. Testing was done at room temperature at a strain rate of about 0.01/min. The size of the crystals varied somewhat but was about 2.5 × 4 × 4 mm. The ends of the compression specimens were parallel to within one degree, hand lapped through 600 grit paper and lubricated with silicone grease.

After mechanical deformation the crystals were examined metallographically, then re-polished, etched and re-examined. Following this they were sectioned with a low speed abrasive saw to produce thin slices which were parallel or nearly parallel to the ⟨100⟩ zone axis and parallel to the direction of loading. Specimens for TEM were prepared from these slices.

RESULTS AND DISCUSSION

A. Two Surface Trace Analysis

Slip was observed on {110}, {112}, {123} and twinning was observed on {332}, {112} and {2, 4.8, 4.8} planes within the limits of uncertainty of this technique The orientations of slip and twinning planes and of deformation products* were determined by two surface trace analysis. The results are summarized in Table I, and the orientation of the twin planes are shown in Fig. 1. Twinning was observed in 9 of the 15 crystals examined. Load drops were usually observed to accompany twinning. The {332} twinning plane is assumed to correspond to the {332}⟨113⟩ twinning system reported by Blackburn and Feeney, although we were not able to determine η_1 from these experiments. The twins were very thin and very numerous, and we were not able to measure the shear or angle of surface offset for any specific pair of striations. The {332} ⟨113⟩ twinning system has been examined in some detail by Richman[5] and by Crocker.[6] The irrational mode {2, 4.8, 4.8} is a permutation of the mode $\{2 i^{-} 2\}\langle 1 \bar{i}^{+} 1\rangle$, $i\pm = \sqrt{8 \pm 2}$, predicted by Bevis and Crocker.[7] In both cases, the shear plane is $(1\bar{1}0)$. The fraction of atoms shearing directly to the twinned position is $\frac{1}{2}$ for the {332}⟨113⟩ mode, whereas it is only $\frac{1}{4}$ for the irrational mode. The more familiar {112} mode was ob-

*In this section it is assumed that all of the deformation products are twins. In section B it is shown that some of these features are actually the result of a martensitic transformation.

J. A. ROBERSON and S. FUJISHIRO are with the Aerospace Research Laboratories and Air Force Materials Laboratory, respectively, Wright-Patterson Air Force Base, Ohio 45433. V. S. ARUNACHALAM is with the Materials Science Division, National Aeronautical Laboratory, Bangalore-17, India. C. M. SARGENT is with the Department of Metallurgy and Materials Science, McMaster University, Hamilton, Ontario, Canada.

Manuscript submitted November 21, 1973.

Table I. Summary of Results

Spec	Type*	Slip	Twin	Load Drip	Stress N/M^2
C2-1	A	–	$1\bar{1}2$	yes	624 (10^6) (a)
C2-2	A	$1\bar{3}2$, 110	$\bar{3}\bar{3}2$	–	–
C3	A	–	$\bar{2}, \overline{4.8}, 4.8$	yes	751 (10^6) (a)
C4	A	123	–	–	D
C1-2	A	110	–	–	D
C1-3	A	321	$\bar{2}, \overline{4.8}, 4.8$	–	D
10	B	112	$\bar{2}, \overline{4.8}, 4.8$	yes	1068 (10^6) (a)
3	B	–	$\bar{3}32$	yes	986 (10^6) (a)
8	B	123	–	no	1020 (10^6) (b)
7	B	123	–	no	979 (10^6) (b)
5	B	112, 123	–	no	958 (10^6) (b)
9	B	–	$\bar{3}32$	yes	1096 (10^6) (a)
4	B	$\bar{1}21$	$\bar{3}32$	yes audible	1034 (10^6) (a)
6A†	B	$\bar{1}\bar{2}3$	233	no	930 (10^6) (b)
6B†	B	112	–	no	930 (10^6) (b)

*A = tension, B = compression, (a) onset of twinning.
†Longitudinal bicrystal, (b) 0.2 pct yield, D = specimen failed in grip.

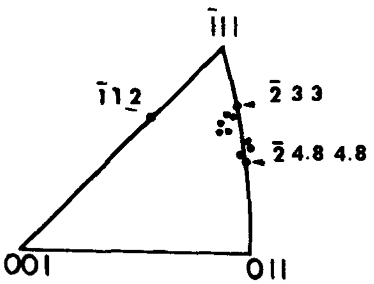

Fig. 1—Twinning planes observed in Beta III Ti alloy crystals.

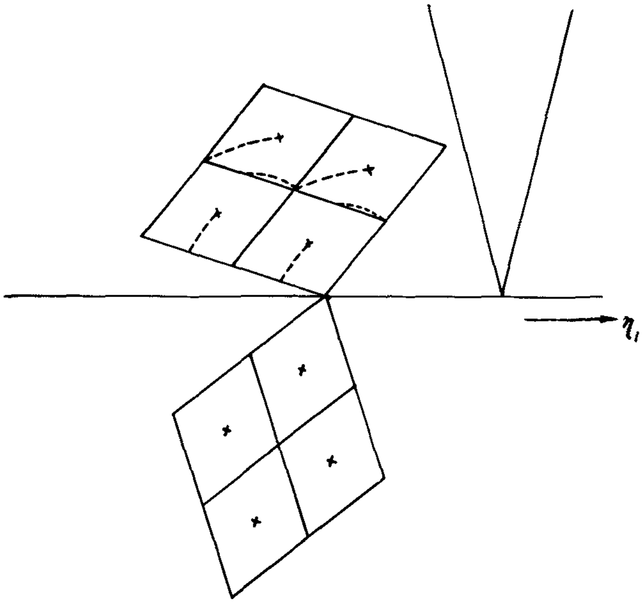

Fig. 2—A shear diagram for $\{2\bar{\imath}^{-}2\}\langle 1\bar{\imath}^{+}1\rangle$ twinning.

served only once in this work and not at all in the work reported by Blackburn and Feeney. These authors report that the passage of a {112} twinning plane could destroy the fine omega precipitates formed in Beta III, as it requires non-cooperative movement of atoms in the omega phase. They suggest that atom movements in the omega phase with the {332} twinning mode are much simpler. A similar explanation is no doubt applicable to the presently reported {2, 4.8, 4.8} mode, although the number of matrix shuffles is greatly increased.

The atomic shears and shuffles required to produce $(2\bar{\imath}^{-}2)[1\bar{\imath}^{+}1]$ twinning are shown in Fig. 2. Two layers of the $(1\bar{1}0)$ plane are shown. In the lower layer alternate rows of atoms shear directly into their final positions. In the second layer, positions indicate by X's none of the atoms shear directly into their final positions. The sum of the shear translations, then, is alternate rows on alternate layers, or $\frac{1}{4}$ as predicted by Bevis and Crocker.[7] The shuffles required to produce the twinned structure are indicated by arrows. Unlike Blackburn and Feeney we are not able to show that omega phase in either of its possible orientations on the $(1\bar{1}0)$ plane would be preserved during the passage of the $(2\bar{\imath}^{-}2)$ interface.

B. Transmission Electron Microscopy

Fig. 3(*a*) and (*b*) represent the surface of a single crystal after 2 pct straining; (*a*) as deformed and (*b*) electrochemically polished to erase slip traces. The

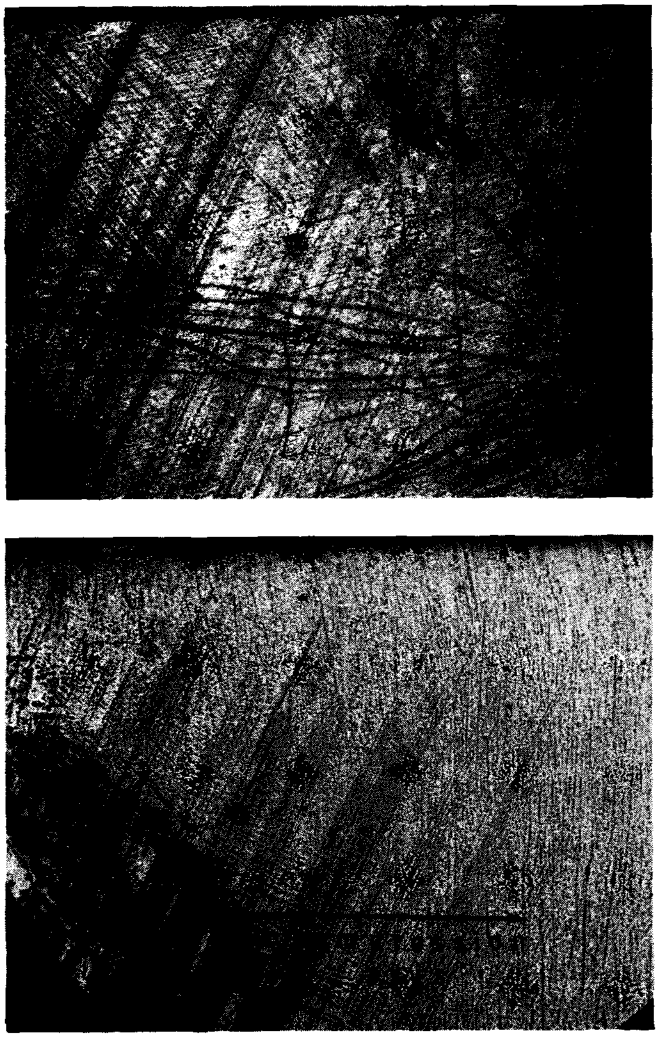
Fig. 3—Optical micrograph of strained crystal: (*a*) as deformed, (*b*) electrochemically repolished to remove slip traces.

surface is within 10 deg of a (001) plane; the direction of compression is indicated by an arrow. The thin films prepared from slices sectioned parallel to the surface were observed by TEM; three different types of twins were found as follows:

{112} type twin. A composite TEM micrograph of bright and dark field of a {112}⟨111⟩ twin is shown in Fig. 4. The beam direction is within 10 deg of the ⟨001⟩ direction. The trace of the composition plane is nearly parallel to the ⟨110⟩ direction. The projection width of the habit plane is about 2800Å. Since the film thickness is in the neighborhood of 2000Å, the habit plane is best interpreted as a ($\bar{1}12$) plane whose pole is inclined 35 deg from the {001} direction. It was difficult to obtain a dark field image of the twin from the (110) reflection at the $s = 0$ condition; a slight tilt of the beam was required to do so. This situation may indicate that the $(110)_T$ plane is not exactly parallel to the $(110)_M$ plane. Such misorientation can be attributed to the high density of dislocations which remain at the composition plane after twinning. In this twin, all the primary diffraction spots from the reflection plane, *i.e.*, the $(212)_T$ plane except the $(\bar{1}\bar{1}0)_T$ spots disappear due to the extinction rule. This condition also contributed to the difficulty in obtaining the dark field image.

{332} type twin. These twins resembled the {112} type twins, *i.e.*, the width ranged from 1 to 2 microns; bundles of them were parallel and had a mean separation of 3 microns. A typical twin of this kind is represented in Fig. 5. The diffraction patterns from both the matrix and the twin are shown as inserts on the appropriate structure.

Fig. 4—Electron micrographs of {112} type twins, matrix zone axis approx. [001]. (*a*) bright field, (*b*) dark field of twin, $g = [110]_T$.

It was found from the pattern analysis that the plane normal for the matrix is close to the $\langle 001\rangle_M$ direction. The zone axis of the twin is near the $\langle 331\rangle_T$ direction. Since the transformation matrix for a (332) twin is not known, there is no way to derive analytically the plane normal of the twin in a specific permutation of $\langle 331\rangle_T$; neither, consequently, can the direction of the trace in the twin be derived analytically. Therefore, it was necessary to deduce the composition plane of the twin by stereographic analysis. All ⟨hkl⟩ trace directions determined from the diffraction pattern which had been indexed in terms of permutations of $\langle 331\rangle_T$ zone axis were plotted on a stereogram. The great circles were drawn with respect to these directions to find whether any of these circles intersected near the prospective major poles with the great circle of the trace direction of the matrix. It is evident from Fig. 6, a segment of the stereogram, that T_0 and T_1 intersect near the ($\bar{3}32$) pole. The projection width of the composition plane and the specimen thickness demonstrates that the angle between the plane normals of the matrix and the composition plane is about 76 deg. This information supports the previous stereographic analysis.

Orthorhombic twin. The results shown in Fig. 7 wer obtained from a specimen which had been strained abou 5 pct in compression. The plane of the foil was parallel to the compression axis. The area shown has been com pletely transformed to orthorhombic martensite which has also undergone deformation twinning. The unit cell is that proposed by Blackburn and Feeney. The observed and calculated interplanar spacings and angles are presented in Table II. The zone axis of the matrix is [$\bar{1}01$], and that of the twin is [$\bar{3}10$]. Twinning has occurred on the (111) plane of the parent martensite. It i. noted that this is a face centered structure and that planes with mixed indices ought not to reflect. The fact that truly forbidden reflections are observed may indicate that the arrangement of the alloying elements in the crystal lattice is ordered to some extent. If this occurs, mixed indices may be observed in spite of the face centered structure.

General comments on the deformation structures. In a report by Rack et al[4] it was cited that spontaneous microtwins have been formed during the thinning procedure of thin foils. This is consistent with the present experiment only if the crystals were not previously deformed. In deformed crystals spontaneous microtwins have never been observed, but slip traces whose

Table II. Interplanar Spacings and Angles in Orthorhombic Martensite*

	Spacings		Angles		
hkl	$d_{Observed}$	$d_{Calculated}$	$h_1k_1l_1$-$h_2k_2l_2$	Obs.	Calc.
002	2.340	2.355	002-132	58	58.4
111	2.340	2.293	002-130	90	90.0
132	1.214	1.227	130-132	32	31.3
030	1.620	1.620	111-030	62	61.8
130	1.432	1.438	111-$\bar{1}2\bar{1}$	105	104.8
$\bar{1}2\bar{1}$	1.780	1.776	030-$\bar{1}2\bar{1}$	43	43.0

*Calculated values based on ABC = 3.12, 4.86, 4.71.[1]

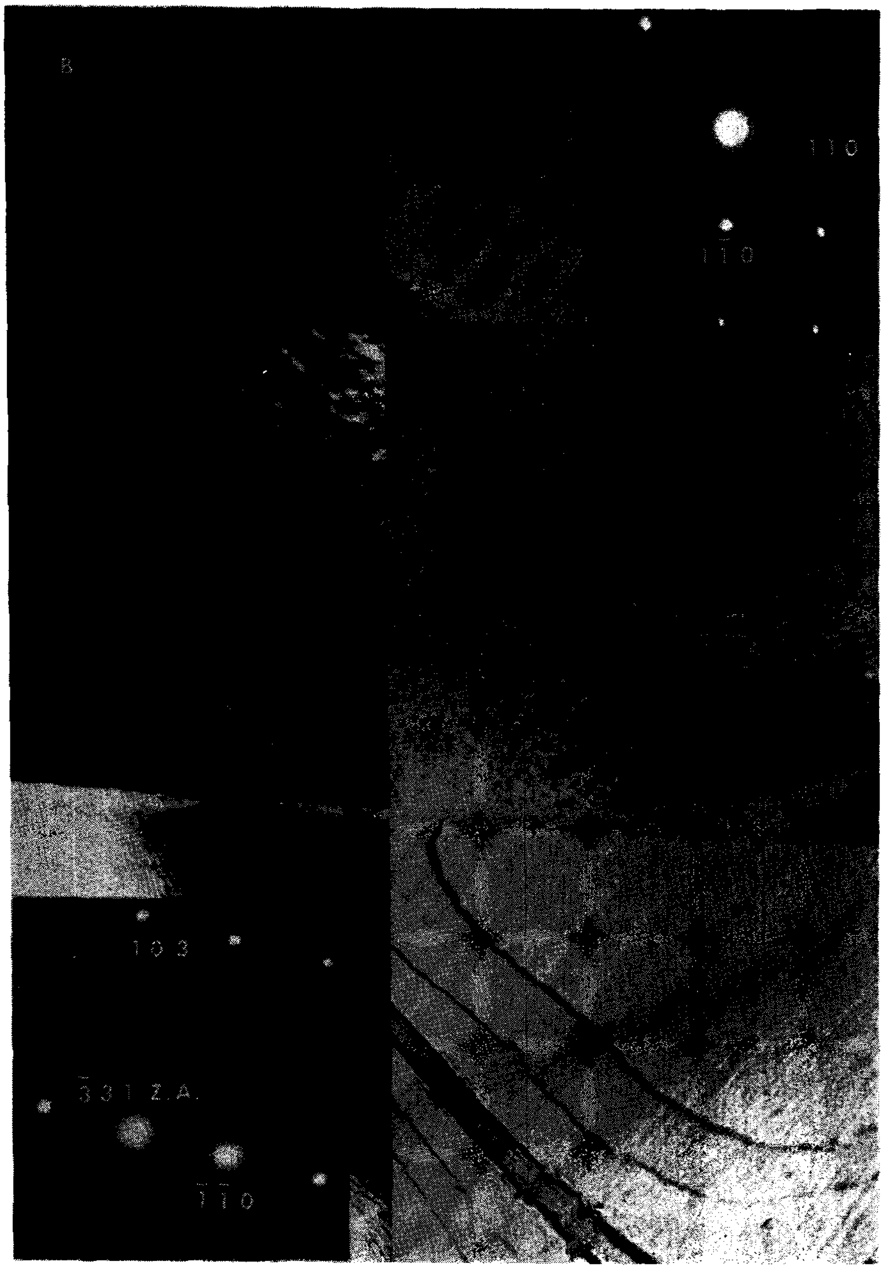

Fig. 5—$(\bar{3}32)$ type twins in Beta matrix. (*a*) Bright field and diffraction pattern from matrix, zone axis ⟨100⟩; (*b*) dark field and diffraction pattern from twin, zone axis ⟨133⟩.

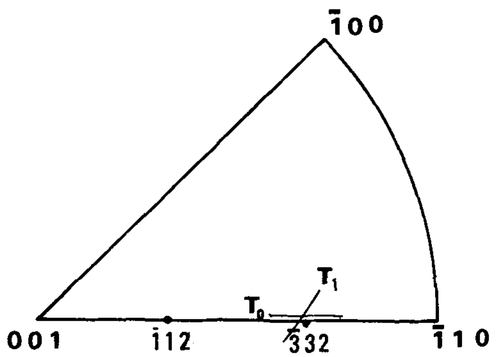

Fig. 6—Stereographic analysis of traces shown in Fig. 5, see text.

appearance is similar to such microtwins were observed. When the foils were tilted appropriately, however, dislocation loops appeared on a coplanar slip plane, {110}; this is shown in Fig. 8.

It seems apparent that after deformation such high density dislocations prohibit the movement of the twinning partials which cause the formation of microtwins. It was concluded from these observations that these microtwins have no connections with the microtwins discussed in the preceding sections. A further explanation of the variance between our results and those of Rack *et al* may be found in alloy chemistry; our alloy was about 1 pct leaner in Mo and 1.5 pct richer in Zr than their alloy was. The yield strength, or twinning stress, of our crystals was abnormally high; audible clicks associated with twinning were emitted by specimen 4 at as high a stress as 1034 (10^6) N/M^2. This strength level probably indicates the presence of a higher volume fraction of omega phase than was present in the material used by Rack *et al.* It is reasonable to expect that a higher volume fraction of omega would be expected to promote twin modes other than {112}.

Athermal omega phase, which gives a mottled appearance as seen in Figs. 4 through 8, is another im-

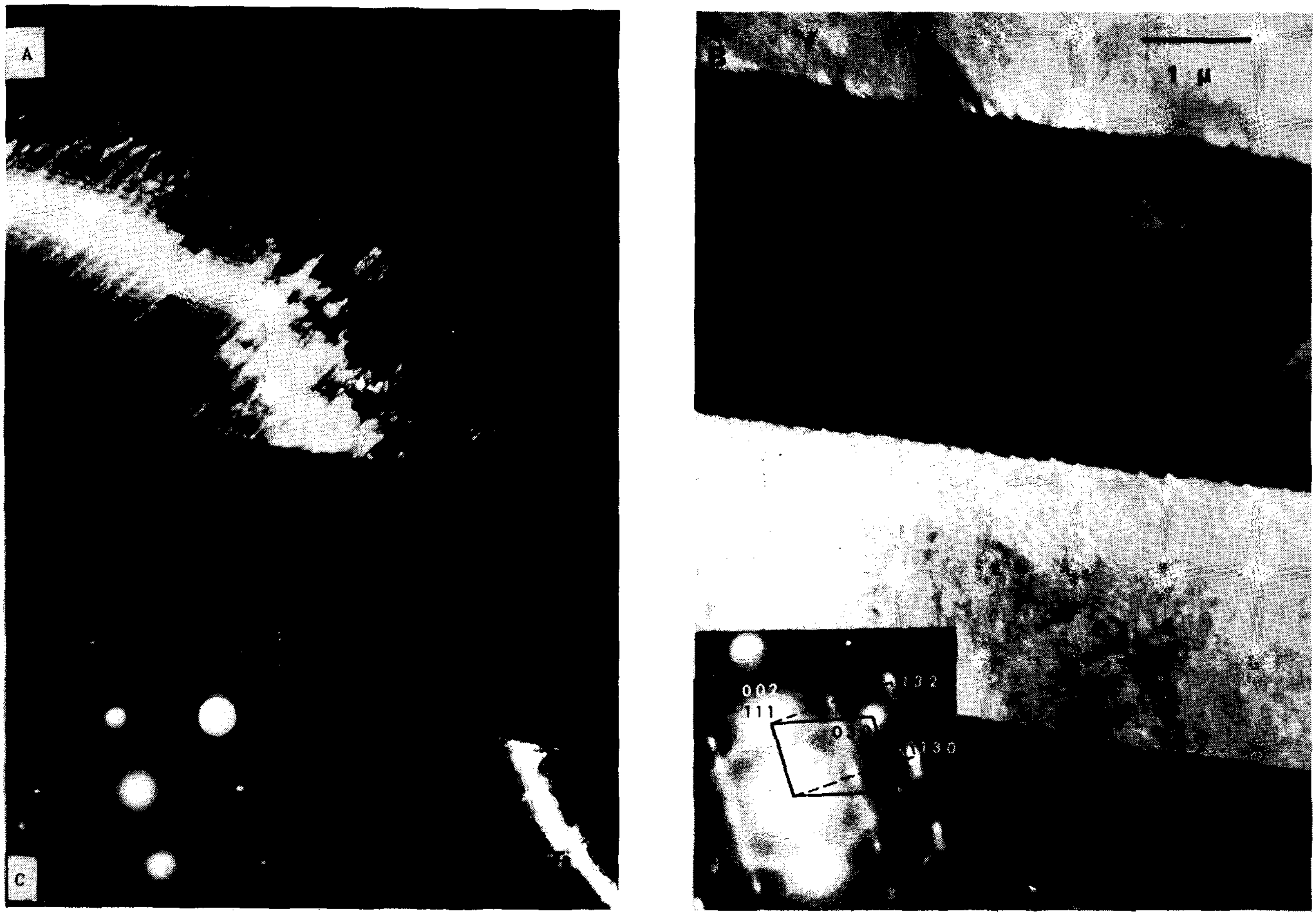

Fig. 7—Selected area diffraction study of orthorhombic martensite. (*a*) Dark field image of matrix, operating reflection $(030)_M$; (*b*) dark field image of both matrix and twins, operating reflections $(\bar{1}\bar{1}1)_M$ and $(00\bar{2})_T$; (*c*) the large bright spot indicates the operating reflection $(030)_M$; (*d*) analysis of stress induced martensite diffraction pattern.

Fig. 8—Slip traces and coplanar dislocation loops in a deformed crystal. These features are similar in appearance but different in structure from "spontaneous microtwins" reported elsewhere.[4]

portant feature of the present alloy. Since this phase produced streaks in the diffraction patterns, defining the spots from the matrix, the twin and the secondary phase was extremely difficult. In order to distinguish these it was necessary to tilt the specimen and to use the dark field technique.

SUMMARY

There is a great deal of controversy regarding the possible deformation structures in Beta III titanium alloy. Many different modes of deformation have been observed and reported. This situation is probably a result of the presence of omega phase in the bcc lattice. This phase suppresses the normal movement of dislocations in the lattice and thereby promotes the occurrence of other modes of deformation. The significance of omega phase is regulated by its size, distribution, and volume fraction; and these characteristics are rather sensitive to composition and thermal history. A great variety of results may therefore be expected. In the present series of experiments slip was observed on $\{110\}$, $\{112\}$, and $\{113\}$, twinning was observed on $\{112\}$, $\{233\}$, and $\{2, 4.8, 4.8\}$. A strain induced martensitic reaction was observed and the martensite also underwent twinning as deformation continued.

ACKNOWLEDGMENT

Mr. T. S. Rowland of the Computer Science Center at Wright Patterson Air Force Base prepared the computer program used in this work. We wish to express our gratitude to him. Copies of the program for computing spacings and angles in orthorhombic structures may be obtained from the authors.

REFERENCES

1. M. J. Blackburn and J. A. Feeney: *J. Inst. Metals,* 1971, vol. 99, p. 132.
2. M. J. Blackburn and J. C. Williams: *Trans. TMS-AIME,* 1968, vol. 242, p. 2461.
3. J. A. Roberson and A. M. Adair: *Met. Trans.,* 1972, vol. 3, 1967.
4. H. J. Rack, D. Kalish, and K. D. Fike: *Mater. Sci. Eng.,* 1970, vol. 6, p. 181.
5. R. H. Richman: *Deformation Twinning,* R. E. Reed-Hill *et al,* eds., Gordon and Breach, New York, 1964.
6. A. G. Crocker: *Acta Met.,* 1962, vol. 10, p. 113.
7. M. Bevis and A. G. Crocker: *Proc. Roy. Soc.,* 1969, vol. A 313, p. 509.