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**Berkeley, California**

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March 1964

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Abstract

Thin sheets of copper and copper aluminum solid solutions were explosively deformed at different pressures and examined in the electron microscope. It was found that after a critical pressure the mode of deformation changed from slip to microtwinning. This critical pressure depends on stacking fault energy.

### Introduction

As yet there have been relatively few electron microscopy investigations of the substructures formed by explosive deformation.<sup>(1,2)</sup> The observations of Nolder and Thomas<sup>(2)</sup> on shock-hardened nickel showed that the dislocations formed a cell structure and that the arrangement of dislocations was much more uniform than after static deformation. A model was proposed on the basis of work hardening<sup>(3)</sup> and twinning<sup>(4)</sup> by which the stacking fault energy was related to the critical pressure for the onset of twinning in shock loaded fcc metals. A relation between twinning stress and stacking fault energy in statically deformed copper alloys has been reported by Venables.<sup>(5)</sup>

The present investigation was undertaken to study the substructures, and in particular twinning, in explosively deformed copper, and copper-aluminum alloys of known stacking fault energies.<sup>(6)</sup>

### Experimental Procedure

Copper of 99.999% purity and copper-aluminum alloys containing 1.10, 2.20 and 8.19 wt. % aluminum were used for this investigation. Specimens of 0.5" diameter and 0.002" thickness were obtained from rolled sheets. After suitable annealing treatments the specimens were explosively deformed at Sandia Corporation<sup>(7)</sup> in an assembly whereby several specimens could be shock hardened at the same time. The specimens were placed at various depths in the assembly with copper plugs as spacer materials between them and were subjected to pressures up to 200 Kbar.<sup>(7)</sup>

Foils for transmission electron microscopic investigation were prepared from deformed specimens by electropolishing in a solution of 33% nitric acid in methyl alcohol at -50°C. The foils were examined in a Siemens Elmiskop I-b

electron microscope operated at 100 kV.

### Results

In all the materials examined the substructures was observed to change from dense dislocation tangles and cells (Fig. 1) to microtwinning with an increase in pressure (Figs. 2 to 5). These results are in qualitative agreement with a previous investigation of explosively deformed nickel.<sup>(2)</sup> The critical value of the pressure required to cause microtwinning was found to depend on the composition of the material, i.e., upon the stacking fault energy.\*

Table I summarizes the results in terms of composition and stacking fault energy  $S$ . The  $S$  value for copper is assumed to be  $70 \text{ ergs/cm}^2$  while the  $S$  values for the alloys are derived from the data of Howie and Swann<sup>(6)</sup>. The lower values of pressure given in column three of the table refer to pressures where no twinning was observed, i.e., where the substructure consisted entirely of cells and dislocation tangles. The cell structure showed a very uniform cell size from 0.1 to 0.3 microns (Figs. 1 and 2a). The upper values refer to the smallest pressures at which twinning was unambiguously detected. Although no deformation between these values were investigated, specimens were examined at lower and higher pressures. In the specimens which showed both cells and twins, it was generally observed that the dislocation density was much smaller than if no twins were present (Fig. 2a and b). In the Cu-8.19% Al alloy twins were observed even in specimens deformed at the lowest pressures investigated (6 Kbar).

\* As will be discussed later the specimen geometry and testing conditions also appear to affect this threshold pressure.

Twinning was always found to occur on  $\{111\}$  planes. From the comparison of Figs. 2b (from 28 Kbar) and 3 (from 200 Kbar), (both from copper), it is seen that the twins are generally much finer and occur in higher densities in the more highly deformed specimens. It is presumably the fineness of these twins that prevented Glass et. al.<sup>(8)</sup> from detecting twinning during an x-ray and metallographic investigation of shock loaded copper. However, recent investigations by metallography<sup>(9)</sup> and x-rays<sup>(10)</sup> have shown the occurrence of twinning in shock loaded copper. The twin densities required for detection by these techniques and hence the pressure values, are comparatively higher than those of our results. This is possibly because the techniques used were not sensitive enough to detect the very earliest stages of twin formation.

Identification of the twins was done by analysis of selected area diffraction patterns and dark field techniques as described previously.<sup>(11)</sup> An example is shown in Fig. 4. Figure 6a shows another micrograph from the same specimen. From its diffraction pattern (Fig. 6b), dark field experiments and trace analyses, it was possible to confirm the presence of  $(\bar{1}\bar{1}1)$  twins.

The density of twins and their fineness was observed to increase with increase in pressure and decrease in stacking fault energy (i.e., higher Al content). This was especially the case in Cu-8.19 % Al alloy ( $S \approx 2.5$  ergs/cm<sup>2</sup>). The fineness of twins produces streaking in the diffraction patterns, e.g., Fig. 5b. The pattern can be explained by twinning as follows. The orientation in Fig. 5b is  $(\bar{1}16)$ , i.e., about 13° away from (001). This pattern agrees with the trace analysis shown in Fig. 5c. The pattern is complicated for two reasons. Firstly, after reflections about the twinning plane  $T_1(\bar{1}11)$ , poles such as (001),  $(\bar{1}\bar{1}1)$  and (111) fall about 7° from the basic circle.



Secondly, since the twins are very fine, the reciprocal lattice is streaked and this gives rise to extra spots on the pattern. The twins seen in the micrograph of Fig. 5a correspond to traces  $T_1$  and  $T_2$  on the stereogram.

In this Cu-8.19 % Al alloy, other complex structures giving streaked diffraction patterns were also observed. In Fig. 7 two such structures are shown. The diffraction patterns for both showed streaking in  $\langle 111 \rangle$  directions. The substructure is too fine to enable one to prove whether they are twins or stacking faults, e.g., the diffraction patterns showed neither shifted nor twin spots.

The explosively deformed specimens always tended to anneal while under observation in the electron beam even though special care is always taken to avoid heating. The polygonized areas thus obtained showed a very fine grain size. This phenomenon is attributed to the very high stored energy resulting from the explosive deformation.

#### Discussion

From our observations it is necessary to consider a twinning criterion, which might account for the changes in mode of deformation in each material. Venables<sup>(12)</sup> has proposed a model for twinning in which the critical shear stress ( $\tau_T$ ) is affected by a stress concentration factor ( $n$ ) and is related to the stacking fault energy ( $S$ ), the Burgers vector of Shockley partial ( $b_1$ ), and to the radius ( $a_0$ ) of a semicircular loop of stacking fault which nucleates the twin according to the expression:

$$n\tau_T = \frac{S}{b_1} + \frac{Gb_1}{2a_0} \quad [1]$$

On the other hand, with increasing plastic deformation the dislocation density increases to some value such that a critical length of the pinned dislocation  $\bar{l}$  is attained (e.g., in the cell walls). It is thought that  $\bar{l}$  should be related to critical cell size.<sup>(2)</sup> The shear stress to bow out such a dislocation is given by

$$\tau = \frac{Gb}{\bar{l}} \quad [2]$$

where G is the rigidity modulus and b the Burgers vector of the unit dislocation. By assuming  $\bar{l}$  to be nearly  $2a_0$ , and comparing these two values of stresses, then, whenever the critical stress  $\tau_c$  exceeds the shear stress for twinning according to equation [1], twinning should be the preferred mode of deformation. Thus, the critical stress for twinning is obtained by eliminating  $\bar{l}$  from equations [1] and [2]:

$$\tau = \frac{Sb}{b_1(nb - b_1)} \quad [3]$$

Presumably the stress concentration factor explains why in some areas of the same crystal twins were observed, while in other areas, regions of high dislocation density were found. No fixed value can be assigned to n, although in some cases Venables<sup>(12)</sup> has suggested  $n \geq 3$  to explain the results of static tension tests.

Two sets of calculations for  $n = 1$  (i.e., no stress concentration), and  $n = 3$  from equation [3] were carried out. The results are shown in Table I. The trend for both sets is in agreement with our observations, i.e., the critical pressure value decreases with decreasing stacking fault energy. A plot for

the calculated values corresponding to  $n = 1$  and the observed values is shown in Fig. 8.

The differences in the two results shown in Fig. 8 can be due to a number of factors one of which is undoubtedly the fact that the calculated values correspond to critical shear stresses, while the experimental values refer to the actual applied stresses. Although at present there are no exact methods for calculating the shear stress from the nominal stress, Davis and Jackson<sup>(13)</sup> have proposed that in the case of shock loading the acting shear stress is about one quarter of the normal stress. Using this correlation our experimental results are seen to be in very good agreement with the calculated values. However, it should be mentioned that the methods for calculating the experimental pressure values are based on a number of assumptions and the values may not be very accurate.<sup>(7)</sup>

Our results are not in quantitative agreement with the results of Nolder and Thomas<sup>(2)</sup> on bulk specimens of nickel, where twinning was observed at and above 350 Kbar. However, the shock loading in the previous work was done on 3 inch dia.,  $\frac{1}{4}$  inch thick plates under different loading conditions.<sup>(2)</sup> Furthermore, according to Munson,<sup>(7)</sup> when exactly the same conditions of deformation and when the same geometry of specimens are used as were employed for the copper experiments described here, the threshold pressure for twinning in nickel was found to be in the neighborhood of 35 Kbar. It was also found by Munson<sup>(7)</sup> that by changing the spacer material between the specimens the threshold pressure for twinning could be raised. It is therefore concluded that the appearance of twinning in the foils depends upon geometry of the specimen as well as the particular shock loading arrangement. Work is currently in progress here as well as at Sandia Corporation to study the effect of thickness of the specimens.

Since the experiments on nickel by Munson<sup>(7)</sup> and those on Cu and Cu-Al alloys were done under identical conditions, it is interesting to make a comparison of the results. As the two threshold pressures for twinning in the thin sheets of Cu and Ni are so close, one expects that the stacking fault energy of the two materials should be nearly the same. From equation [3] we obtain:

$$\tau = \frac{S}{a(0.41n - 0.236)} \quad [4]$$

where  $a$  is the lattice parameter. As  $a$  for the two metals (Cu and Ni) is nearly the same (3.615 and 3.542 Å respectively),  $\tau$  can be taken as directional proportional to  $S$ . Using this equation and Fig. 8, and finding  $S$  corresponding to 35 Kbar,<sup>(7)</sup> we would predict that  $S \sim 80$  to 100 ergs/cm<sup>2</sup> for nickel, a result which is in very good agreement with that reported by Thornton and Hirsch.<sup>(14)</sup> It should be emphasized that such correlations are possible only when the experiments are always carried out under identical conditions. Changing the conditions of the test undoubtedly changes  $n$  and hence  $\tau$  (equation [4]).

Finally, mention may be made about an effect observed in the Cu-8.19% Al alloy. Preliminary micrographs suggest that a phase transformation, similar to the normal martensitic structures reported in more concentrated Cu-Al alloys by Swann and Warlimont,<sup>(15)</sup> has been observed at and above 27 Kb pressure. This effect is similar to the transformation in iron<sup>(1)</sup> and is under further investigation.

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Table I

% Al	SF Energy <sup>(5,6)</sup> (s)	Deformation pressure range in Kbar for critical twinning pressure	Calculated twinning shear stress and critical $\bar{l}$			
			For n = 1		For n = 3	
			Shear stress in Kbar	$\bar{l}$ in $\mu$	Shear stress in Kbar	$\bar{l}$ in $\mu$
0	70	16-28	11.1	0.01	2	0.06
1.10	50	12-15	8.0	0.021	1.4	0.13
2.20	35	6-12	5.5	0.03	1	0.18
8.19	2.5	< 6	0.4	0.42	0.07	2.6

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Figure Captions

Fig. 1. Transmission electron micrograph from Cu-1.10 % Al alloy after deformation at 15 Kbar. The area shows dislocation tangles arranged in cell structure.

Fig. 2. (a) Cell structure and (b) twinning observed in copper after deformation at 28 Kbar. The orientation of the foil in (b) is (001).

Fig. 3. Showing twin structure in copper explosively deformed at 200 Kbar; notice the fringe contrast and preferential etching of twinned areas. The foil is in (001) orientation and the twins are on {111} planes.

Fig. 4. (a) Cu-1.10 % Al alloy after 27 Kbar. The twins lie along the (111) plane, the foil orientation being (123). (b) Dark field micrograph of a twin diffraction spot showing the contrast reversal of (111) twins.

Fig. 5. (a) Cu-8.19 % Al alloy after 68 Kbar. The diffraction pattern (b) shows that the structure is twinned (see text for explanation). (c) The trace analysis of (a) and (b).

Fig. 6. (a) Cu-1.1 % Al alloy after 27 Kbar showing twins and dislocations. The foil orientation is (013) and the twins lie along the traces of the (111) planes. (b) The diffraction pattern shows overlapping matrix and twin spots. Contrast reversal at twins occurs in the dark field image of spot A but not in the dark field images of spots B and C.



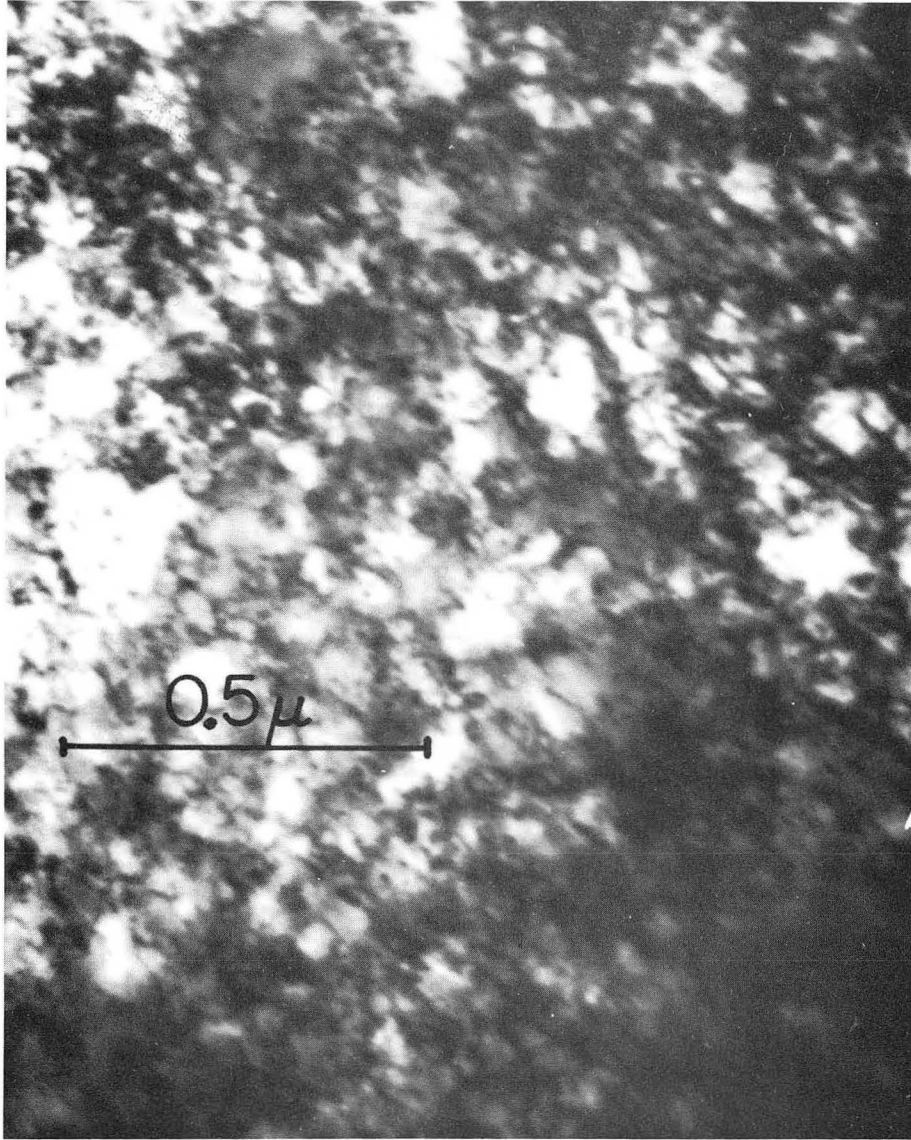
Fig. 7. Cu-8.19 % Al alloy deformed at (a) 68 Kbar and (b) 128 Kbar.

Both micrographs show very high dislocation density and faults.

The faults lie along the traces of {111} planes and cause streaking of the diffraction patterns along corresponding  $\langle 111 \rangle$  directions.

Orientation in both figures is [110].

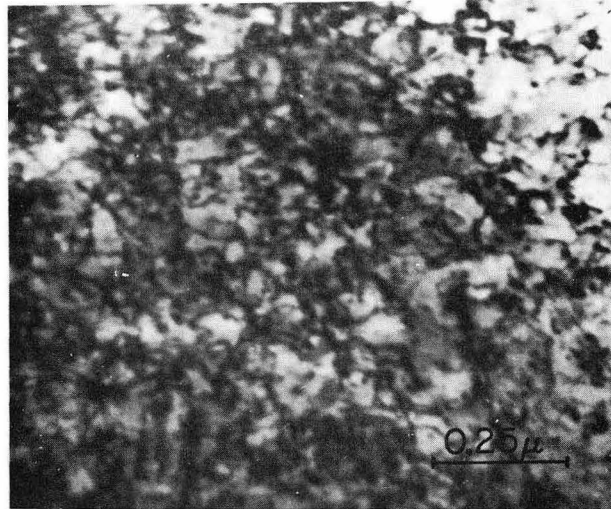
Fig. 8. The calculated critical shear stress value for  $n = 1$  is plotted for comparison with the observed pressure ranges which cause twinning.



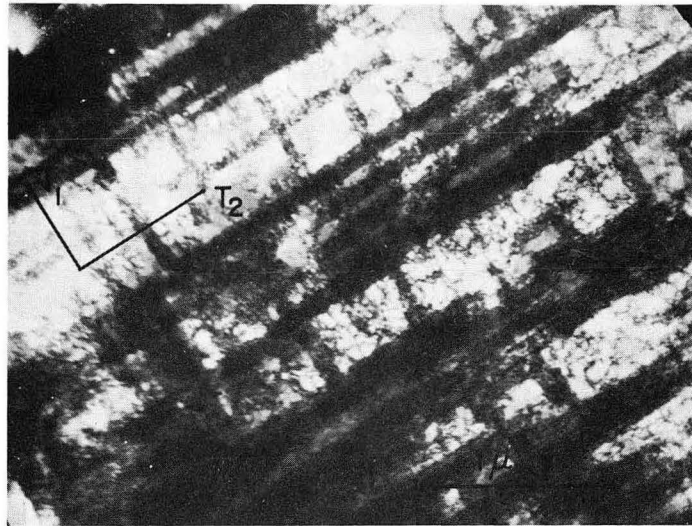
ZN-4242

Fig. 1

(a)



(b)



ZN-4373

Fig. 2

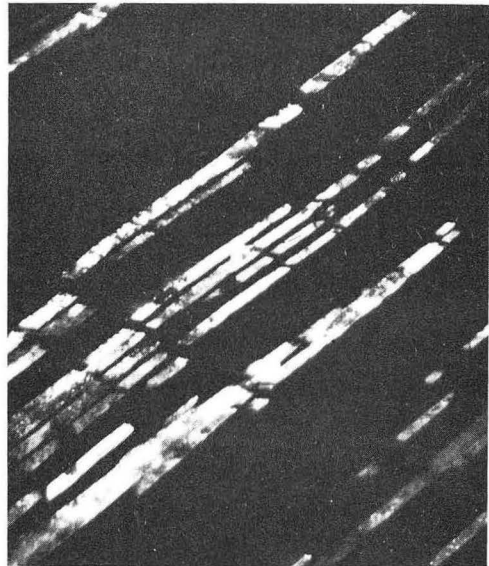


ZN-4244

Fig. 3



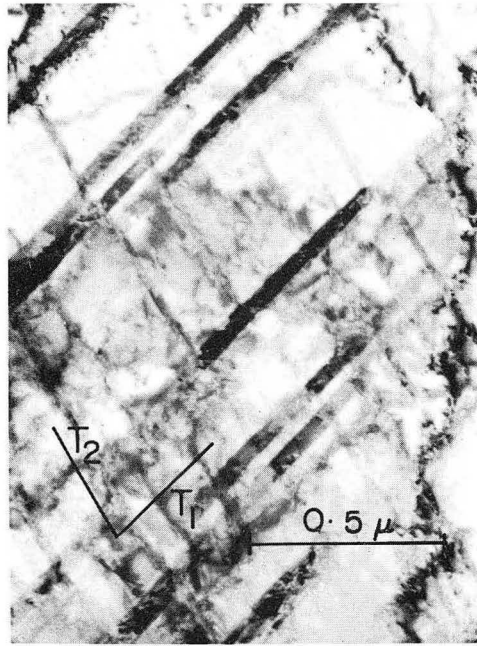
(a)



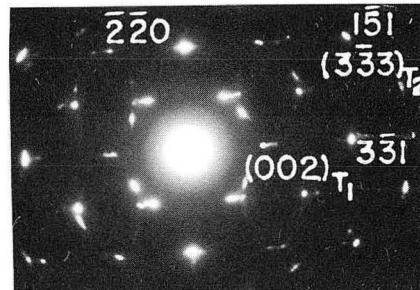
(b)

ZN-4245

Fig. 4



(a)

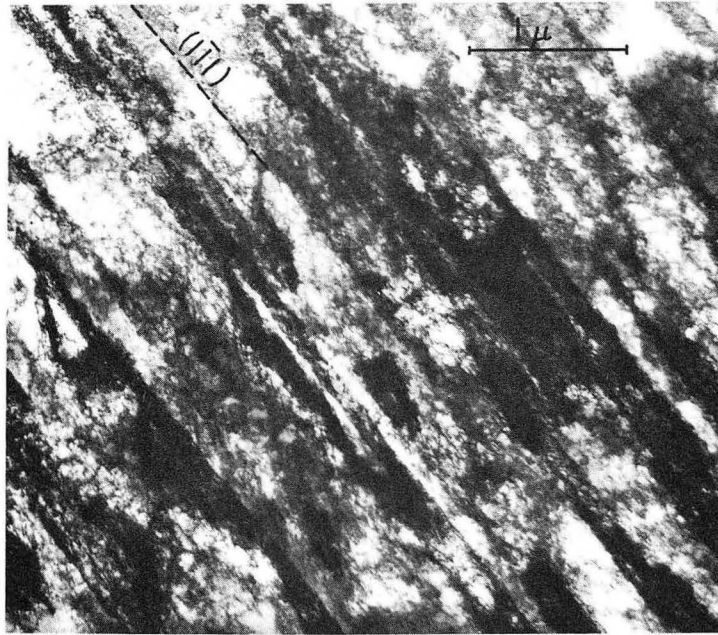


(b)

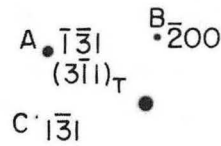
ZN-4246

Fig. 5a b





(a)

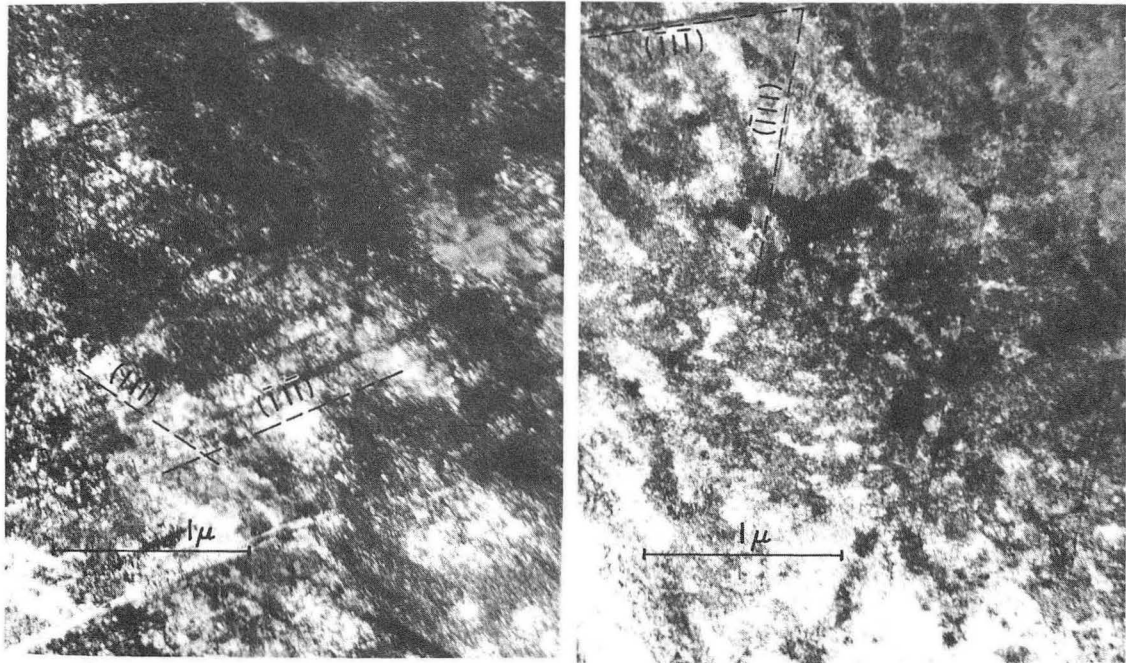


(b)

ZN-4247

Fig. 6



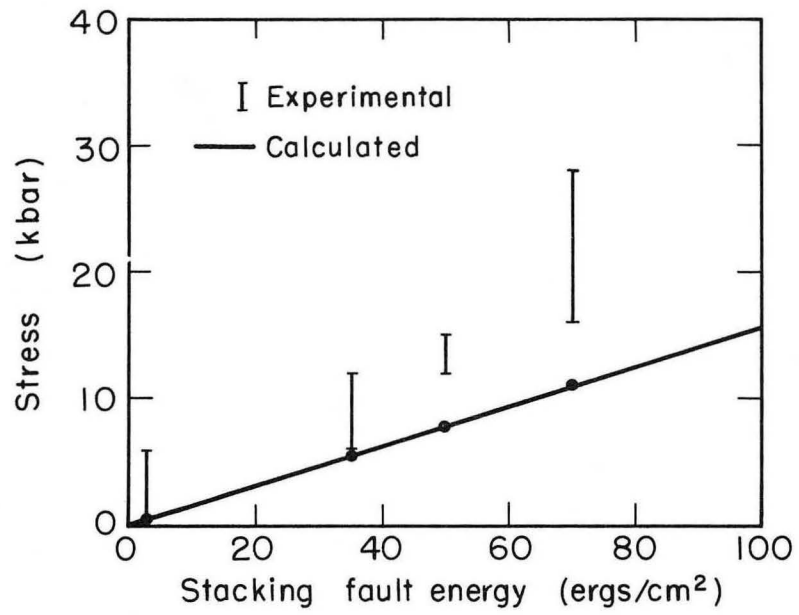


(a)

(b)

ZN-4248

Fig. 7



MU-33510

Fig. 8

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