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Observations on Twinning in Zone-Refined Tungsten

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Mechanical twins were produced in zone-refined tungsten single crystals by explosive working at room temperature. These twins are parallel to {112} planes and have irregular boundaries rather than the classical plane twin boundaries. These boundaries are grooved surfaces in which the grooves themselves are parallel to a <111> direction and the sides of the grooves appear to be parallel to {110} planes.

TWINS were produced in tungsten single crystals by explosive working at room temperature. These twins differ in character from any previously reported for tungsten; however, they are similar to those found in molybdenum after compression at −196°C.[1]

Deformation twins "resembling Neumann bands in ingot iron" have been observed in tungsten by Bechtold and Shewmon.[2] This observation was made with sintered polycrystalline tungsten pulled in tension to fracture at 100°C and using a strain rate of 2.8×10^{-4} sec^{-1}. More recently Schadler[3] found deformation twins in zone-refined tungsten single crystals pulled in tension at −196° and −253°C. These tests were conducted using a strain rate of 3.3×10^{-4} sec^{-1}, and the twin bands were found to be parallel to a {112} plane.

Deformation twins in tungsten's sister metal, molybdenum, were observed by Cahn.[4] These twins were produced by compressing small (0.7 mm) vapor-deposited molybdenum single crystals at −183°C. The compression was performed "by impact." By the use of precession X-ray techniques, Cahn was able to identify the twin plane as {112} and the shear direction as <111>.

Mueller and Parker[1] produced deformation twins in polycrystalline electron-beam-melted molybdenum by compression at −196°C. Their "loading rate" was 5000 psi per min which, judging from their stress-strain curve, corresponds to a strain rate of approximately 0.3×10^{-4} sec^{-1}. These twin bands were found to be parallel to {112} planes; however, they differed in appearance from previously observed twins. In place of straight and parallel twin boundaries they were found to be irregular, jagged, and sawtoothed. The sides of the saw teeth were identified as {110} planes and irrational planes of a {111} zone. The twins observed in the present work in tungsten single crystals are similar in appearance to those of Mueller and Parker in polycrystalline molybdenum.

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EXPERIMENTAL PROCEDURE

The starting material used in this investigation was 3/16-in. diam commercial tungsten rod produced by powder-metallurgy techniques. This material was converted to a single crystal by the electron-bombardment floating-zone technique.[5] The process was carried out in a vacuum of 10^{-5} mm of Hg using a traversing speed of 4 mm per min.

Segments (≈2 in. long and 3/16 in. in diam) of two crystals (A and B) produced in this manner were studied. Crystal A received one zoning pass, while crystal B received two passes.

The two crystals were explosively worked at Battelle Memorial Institute in the following manner. A 1/2-in.-thick layer of plastic was applied to the crystals to serve as a buffer in an attempt to prevent cracking. The composite, crystal and buffer, was then wrapped with 1/8-in.-thick DuPont sheet explosive EL506A2 and detonated in water at room temperature.

Metallographic samples of the worked crystals were prepared, and back-reflection Laue X-ray patterns were obtained using unfiltered molybdenum radiation.

RESULTS AND DISCUSSION

Blasting the crystals as described above failed to prevent cracking. The crystals fractured into several fragments about 3/16 to 1/2 in. long; however, the fragments were of sufficient size to be useful for the subsequent study.

The diamond pyramid hardness of the crystals after blasting was in the range 430 to 450 as compared with 340 for the as-melted material, which shows a definite hardening resulting from plastic deformation. These hardness values were obtained using a 1000-g load and taking readings only in sound portions of the crystals free of cracks.

The crystals exhibited profuse twinning as shown in Fig. 1. No such structure is present in the as-melted condition. Most of these twins have jagged twin boundaries and are similar in appearance to those found in molybdenum by Mueller and Parker. The twins in both crystals were found to be parallel to {112} planes. This identification was made by using the conventional two-trace method.

Subsequent efforts to describe these twins more fully were carried out on crystal A. If the longitudinal axis of crystal A is placed in the (001)-(011)-($\bar{1}$11) basic triangle of the standard cubic stereographic projection, as in Fig. 2, then the two sets of twins shown in Fig. 1 are parallel to the (112) and (121) planes.

Fig. 3 shows a schematic representation of a twin with jagged boundaries. This type of twin with a <111>

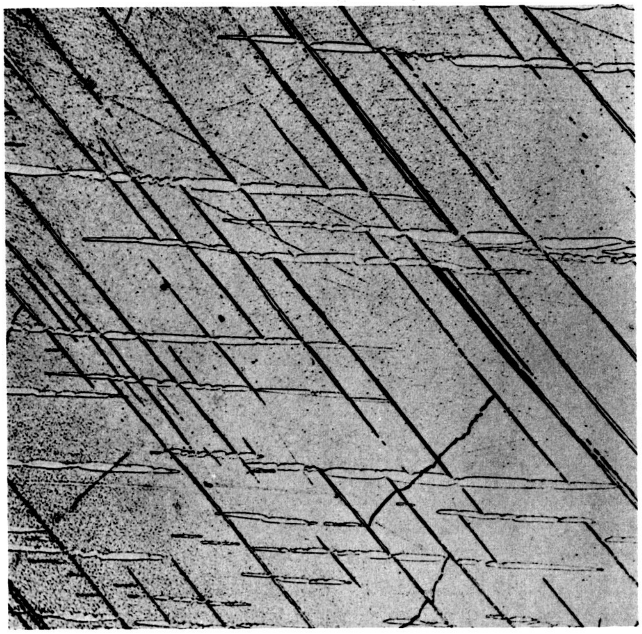

Fig. 1—Transverse section of crystal A. Etchant, Murakami's reagent. X250. Reduced approximately 44 pct for reproduction.

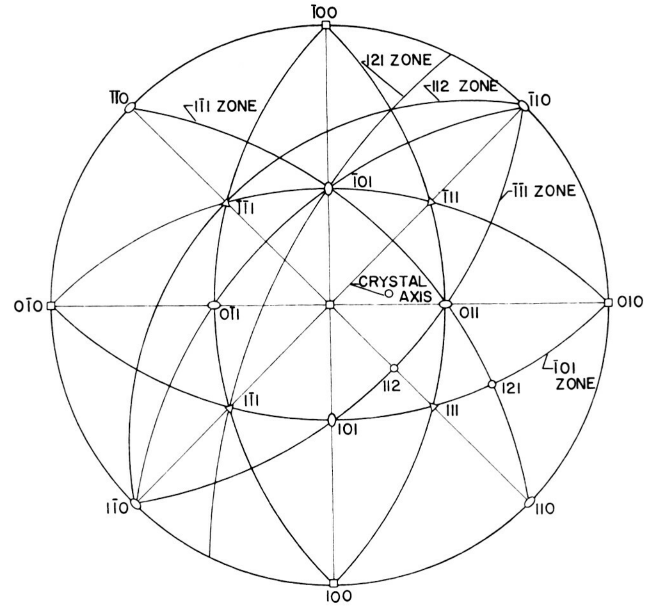

Fig. 2—Crystal A located in (001) standard projection.

shear direction is the type proposed by Mueller and Parker for molybdenum. It is apparent from Fig. 3 that a cut perpendicular to the shear direction would yield jagged twin boundaries and a cut parallel to the shear direction would yield straight twin boundaries. More generally, exposing any plane in the zone of the shear direction should result in straight twin boundaries (assuming the grooves of the twin surface are continuous as depicted in Fig. 3), while any plane not in this zone should result in jagged boundaries.

In this work the type of twin depicted in Fig. 3 with a <111> shear direction was assumed, and the

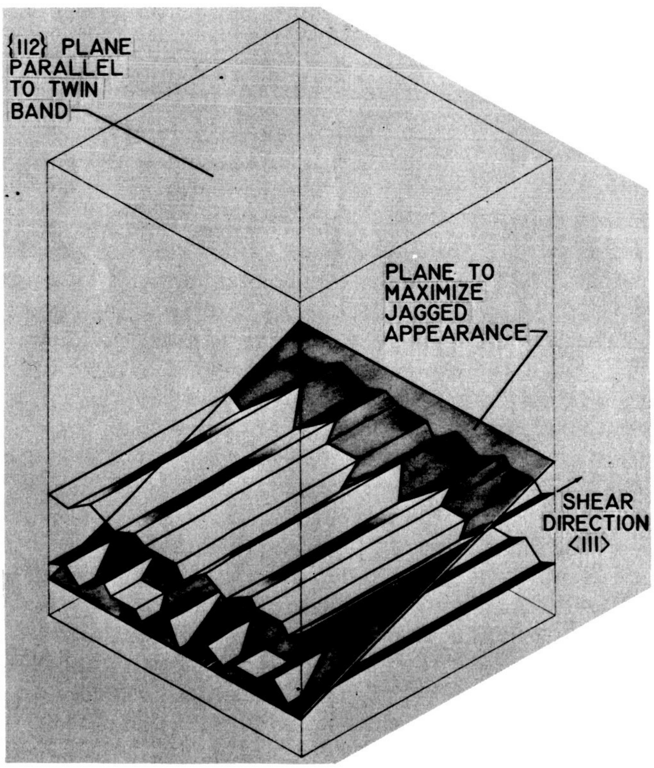

Fig. 3—Schematic view of twin showing irregular twin boundaries.

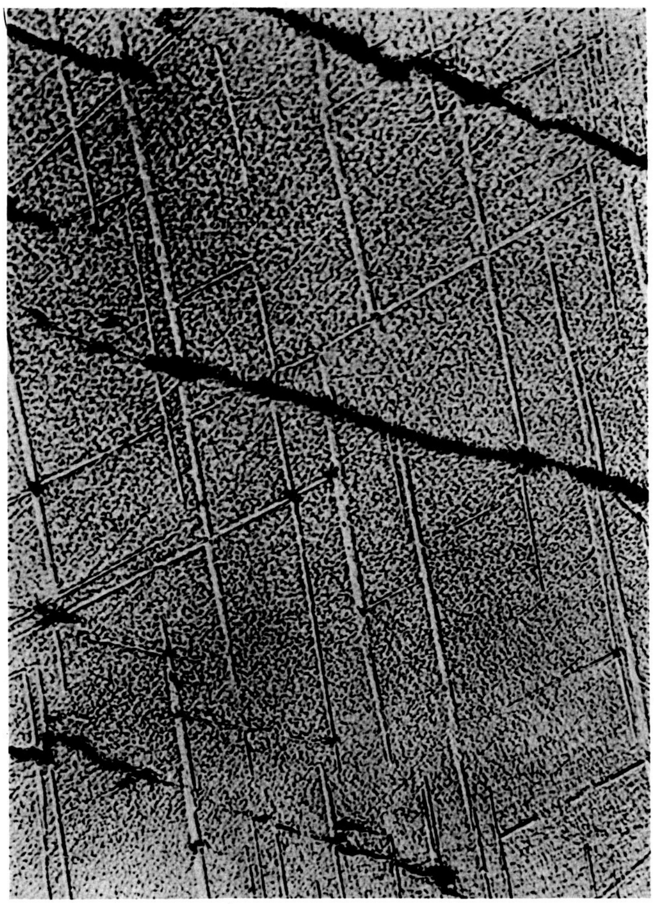

Fig. 4—(011) plane of crystal A showing straight twin boundaries. Etchant, Murakami's reagent. X650. Reduced approximately 40 pct for reproduction.

crystal was then cut to expose planes which should result in straight and jagged boundaries in an effort to verify or disprove the assumed twin.

As pointed out above, any exposed plane in the zone of the shear direction should exhibit straight twin boundaries. In a case such as crystal A, where two sets of twins are present, there is only one plane that would show straight boundaries for *both* sets of twins, and that plane is the plane common to both shear-direction zones. Since the two sets of twins present in crystal A were found to be parallel to the (112) and (121) planes, their assumed shear directions are $[\bar{1}\bar{1}1]$ and $[1\bar{1}1]$ respectively. The plane common to both the $(\bar{1}\bar{1}1)$ zone and the $(1\bar{1}1)$ zone is (011); thus, exposing the (011) plane should result in straight twin boundaries for both sets of twins.

Fig. 4 shows the (011) plane of crystal A, and indeed both sets of twins exhibit straight boundaries as predicted. The fact that all the twin boundaries of Fig. 4 are straight verifies that the grooves of the twin surface are continuous. The two sets of twins shown in Fig. 4 are approximately 70 deg apart, as they should be, since the (112) and (121) planes intersect the (011) plane in the $[\bar{1}\bar{1}1]$ and $[1\bar{1}1]$ direction, respectively, which are 70.5 deg apart.

In order to identify the components of the sawtoothed twin boundary, *i.e.*, the sides of the saw teeth, it was desirable to expose a plane which would maximize the jagged character of the twin boundary. It is apparent from Fig. 3 that this plane should be near the plane which is parallel to the twin band, *i.e.*, $\{112\}$, and on the zone connecting this plane and the shear direction. In the case of crystal A with its two sets of twins, exposing the (111) plane is a compromise to maximize the jagged character of the boundaries of both sets of twins. In order to show a maximum jagged appearance in the boundary of the twins parallel to the (112) plane, a plane of the $(\bar{1}10)$ zone near (112) should be exposed. Similarly, a plane of the $(\bar{1}01)$ zone near (121) should be exposed in order to maximize the jagged appearance of the boundary of the twins parallel to the (121) plane. The plane which represents a compromise between these two conditions is located at the intersection of the $(\bar{1}10)$ and $(\bar{1}01)$ zones, *i.e.*, the (111) plane.

The (111) plane of crystal A is shown in Fig. 5. Both sets of twins exhibit the irregular boundaries.

The lengths of the sides of the saw teeth, as shown in Fig. 5, are short, and thus the angular measurements between these and the twin band are inaccurate. The only thing that can be said for these measurements is that, in the main, the angle between the side of a saw tooth and the twin band is in the neighborhood of 60 deg. This suggests that the components of the saw teeth are planes of the $\{110\}$ type.

The two sets of twins shown in Fig. 5 are 60 deg apart, as they should be, *i.e.*, the (112) and (121) planes intersect the (111) plane in the $[\bar{1}10]$ and $[\bar{1}01]$ directions, which are 60 deg apart.

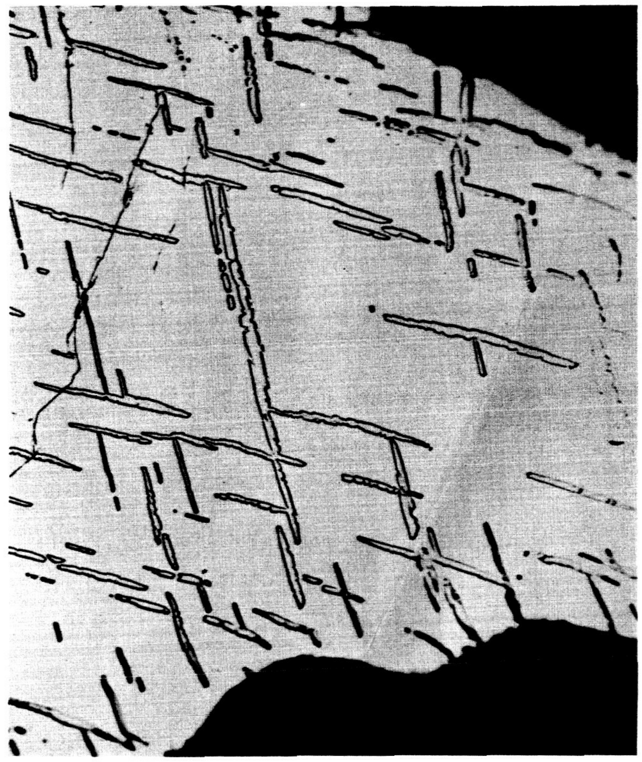

Fig. 5—(111) plane of crystal A showing jagged twin boundaries. Etchant, Murakami's reagent. X650. Reduced approximately 48 pct for reproduction.

CONCLUSIONS

As a result of this work the following conclusions may be drawn:

1) Zone-refined single-crystal tungsten will deform plastically by mechanical twinning at room temperature when subjected to explosive loading.

2) The twins so formed have irregular boundaries rather than the classical plane boundaries and are parallel to $\{112\}$ planes.

3) The irregular twin boundaries are grooved surfaces in which the sides of the grooves appear to be parallel to $\{110\}$ planes and the grooves themselves are parallel to a $\langle 111 \rangle$ direction.

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