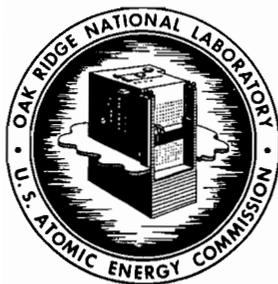


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THE EFFECT OF IMPURITIES ON MECHANICAL TWINNING AND
DISLOCATION BEHAVIOR IN BODY-CENTERED CUBIC METALS

J. O. Stiegler
C. J. McHargue

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ABSTRACT

Mechanical twins have been observed after low-temperature deformation of pure body-centered cubic metals and after room-temperature deformation of certain solid solution alloys. In both cases the presence of interstitials inhibited the twinning process or transferred it to lower temperatures. In this paper, dislocation substructures, as observed by transmission electron microscopy, are reported for materials which twin during deformation and are contrasted with substructures in materials which fail to twin. Differences are interpreted in terms of an increased lattice frictional stress and a reduced ability of dislocations to cross slip in high alloys or during low-temperature deformation. Mechanisms by which these effects alter the twinning behavior are discussed. There is evidence that the stacking fault energy is not lowered by the alloying action. It is further argued that stress concentrations resulting from the altered dislocation configuration are responsible for the enhanced twinning in the alloys. In addition, a raised cleavage strength and increased ability to hold interstitials in solution may act to prevent brittle fracture. The inhibiting effect which interstitials exert on twinning is explained in terms of interstitial precipitates providing sites where dislocation tangles may form and thus break up potential centers of stress concentration.

Transmission electron microscope observations of a Cb-40 wt % V alloy are described which show that the twins generally run entirely across the grains in lightly deformed materials. Twins which end within grains have heavy dislocation tangles at blunt ends. Notches in twins are produced either by slip dislocations from an external source cutting through a twin or by slip dislocations originating in the twin itself and cutting through a coherent boundary. Twins are nucleated in a grain by other twins in an adjacent grain intersecting their common boundary or by

stress concentrations produced by the dislocation substructure. Dislocations, which are probably originally generated at the grain boundaries, appear to multiply by a cross slip mechanism.

These observations suggest that solid solution alloys of the refractory metals can be developed which have superior strength to the pure metals and, because of their ability to twin, may be fabricated readily at room temperature.

INTRODUCTION

Much conflict appears in the literature on mechanical twinning in body-centered cubic metals and alloys due principally to impurity effects. Under appropriate deformation conditions, however, all the body-centered cubic transition metals have been observed to twin.¹ In general, low temperature of deformation, high purity with respect to interstitials, and high rate of deformation all favor twinning in the pure metals.²

McHargue and McCoy² systematically studied the effect of type and quantity of interstitial elements on twinning behavior of columbium at liquid-nitrogen temperature by slow or impact deformation. For slow deformation, 1500 to 1900 ppm O₂, 300 to 500 ppm N₂, and 150 to 200 ppm C were observed to suppress twinning completely. Hydrogen did not appear to affect the twinning behavior. Twins were observed at all levels of deformation upon impact loading although cleavage cracking occurred in the more impure specimens. The measured concentrations of interstitials necessary to suppress twinning were about twice the estimated lattice solubilities after slow cooling,³ which suggests that interstitial precipitates possibly at dislocations were responsible for the observed effects.

Recently some solid solution alloys of molybdenum-rhenium,⁴ tungsten-rhenium,⁴ and columbium-vanadium⁵ have been developed which twin readily at room temperature under slow deformation rates. The reason for the enhanced twinning behavior of the alloys is unknown, but it has been suggested⁴ that, as occurs in face-centered cubic alloys, the stacking fault energy is lowered by the alloying action which aids in the nucleation of stable twins.

Hobson and McHargue⁵ made a thorough study of the effects of composition and temperature on mechanical twinning in the columbium-vanadium system. Using a Rockwell hardness tester equipped with a constant temperature bath to apply a reproducible load, they were able to survey twinning behavior as a function of composition between liquid-nitrogen temperature and about 200°C. By using an audibility test to detect twinning, which subsequent metallographic examination showed to be completely reliable, they further were able to define a twinning transition temperature below which twinning was the important mode of deformation and above which twinning ceased to occur. The remarkable feature of this investigation was the extreme sharpness of the transition region (10-40°C) as is illustrated in Fig. 1. This behavior was extremely reproducible from specimen to specimen and on raising as well as lowering the temperature of deformation. These authors also observed that the presence of oxygen lowered the twinning transition temperature as is shown in Fig. 2.

In understanding the mechanisms by which interstitial impurities inhibit twinning and certain substitutional impurities enhance it, one must consider the mechanisms of the nucleation and growth of a twin. The lack of a well-defined, reproducible critical resolved shear stress for twinning strongly suggests that internal stress concentrations are necessary to nucleate a twin, either to unlock a twinning mill⁶ or to create twinning partial dislocations⁷ homogeneously. The most elegant demonstration that the latter is the case was given by Price⁸ in some experiments in which thin, dislocation-free platelets of zinc were twinned in the electron microscope while under observation. Price observed that twins were nucleated at centers of stress concentration, such as corrosion pits or reentrant corners, and further, by taking into account the geometrical stress concentration factor, he was able to calculate a critical resolved shear stress for twinning, which was independent of crystal size, of about 50 kg/mm². No dislocation pole was ever observed; the twinning partials were nucleated by the stress concentration. Fourie, Weinberg, and Boswell⁹ observed similar behavior in electrothinned tin crystals; and in bulk zinc crystals, Bell and Cahn¹⁰ observed that twinning did not occur until double slip had been initiated, evidently to produce pileups or other obstacles which were able to act as stress concentrators.

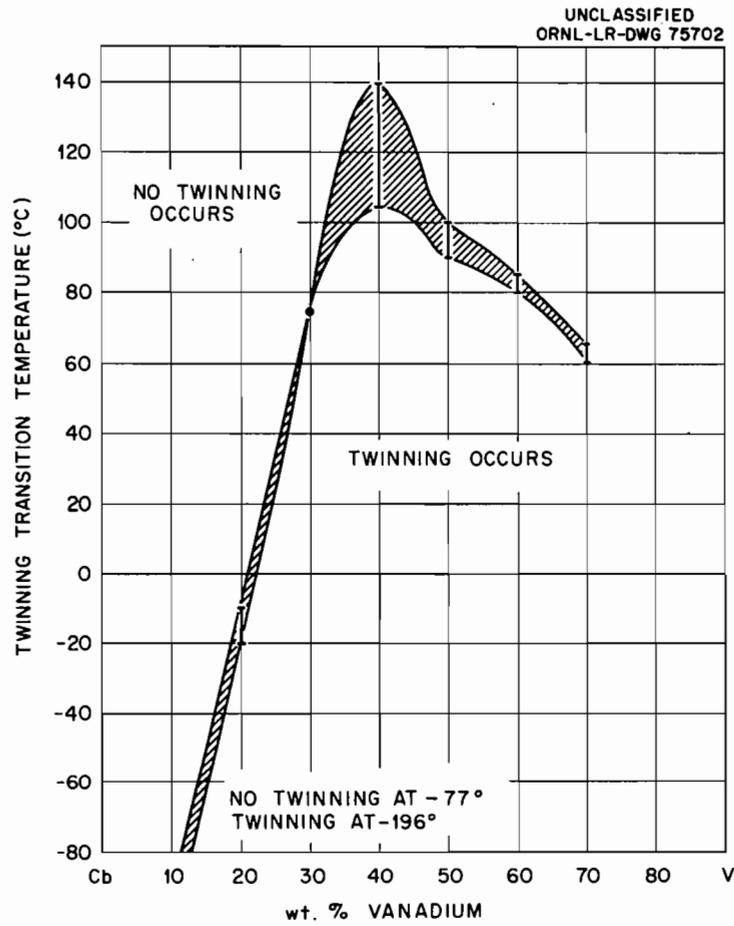


Fig. 1. The Effect of Composition and Temperature on Mechanical Twinning in the Columbiu**m**-Vanadium System.

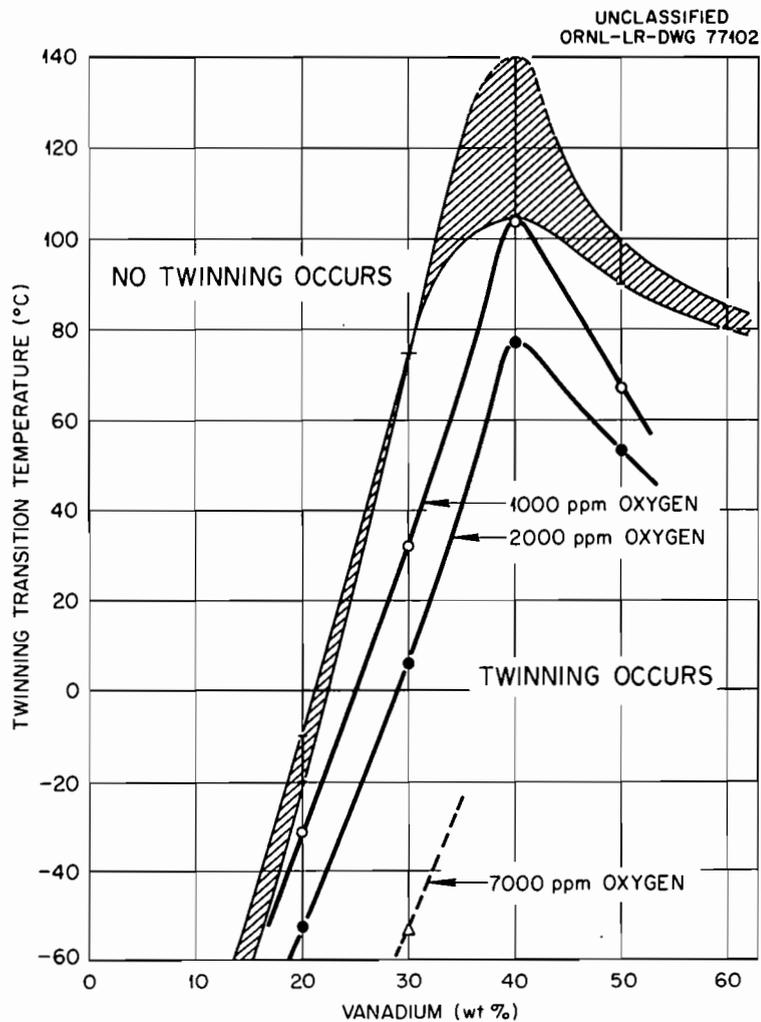


Fig. 2. The Effect of Oxygen Concentration on the Twining Transition Temperature of Columbium-Vanadium Alloys.

It becomes obvious therefore that, in seeking to understand the reasons for enhanced twinning behavior in substitutional solid solutions and the inhibited twinning behavior in interstitial solutions, it is necessary to examine the ways in which the impurities influence slip or other processes which might supply possible sites for stress concentration. In this paper, observations are reported of a transmission electron microscope study of columbium and columbium-vanadium alloys under conditions in which twins both were and were not formed. Differences in substructure are related to the twinning mechanism, and a detailed discussion of stacking fault energy in the alloys is given. In addition, structural features of the twins are described, and information on the nucleation of twins and the multiplication of dislocations is presented.

EXPERIMENTAL PROCEDURE

Arc-melted ingots of columbium-vanadium alloys containing 20 and 40 wt % V and electron beam-melted crystals of high-purity columbium were rolled to strips 0.010 in. thick with intermediate anneals where necessary. They were given a final vacuum anneal at 1000-1200°C for 2 hr prior to deformation by cold rolling, tension, or bending. After this treatment they contained no more than 300 ppm interstitials. Electron microscope specimens were prepared either by chemical polishing in solutions of nitric and hydrofluoric acids or by electropolishing in a solution composed of 9 parts sulfuric acid to 1 part hydrofluoric acid using carbon electrodes in the Bollman manner. The foils were examined in a Hitachi HU-11 electron microscope operated at 100 kv using the biaxial tilting stage.

RESULTS AND DISCUSSION

Dislocation Substructures

Slip dislocations produced by light deformation were observed to exist in groupings that were characteristic of the composition of the alloy and the temperature of deformation. Distinct substructures were produced which characterized materials that twinned during deformation

from those that did not. Furthermore, the individual dislocations in the two substructures reacted differently to stresses set up by the electron illumination.

The well-known cell structure developed early in the deformation process in pure columbium single crystals as well as polycrystals after deformation by either slow compression or cold rolling at room temperature, as is illustrated in Fig. 3. That is, the dislocations interacted with one another to form three dimensional tangled walls surrounding cells that remained relatively free of dislocations. A few small loops were formed, probably by one of the cross slip mechanisms,¹¹ but after about 10% reduction in thickness most of them had been cleared out so that the cell structure was the prominent feature. The sizes and shapes of the cells varied somewhat from specimen to specimen as a result of differences in orientation. Nevertheless, no regions were observed in which the cell structure was not evident.

Previous work had shown that deformation twins could not be produced in columbium of this purity by slow deformation at room temperature. This was verified in this study in which no twins were observed except in one isolated instance near the surface of a specimen where a high stress concentration may have been present during rolling.

In contrast, no evidence of a cell structure was observed in the Cb-40 wt % V alloy which twinned readily at room temperature after deformations of up to 30% by rolling. Typical dislocation structures after various deformations are shown in Figs. 4-7. These micrographs were taken of specimens which were heavily twinned. The areas shown in the figures, however, are removed from the twins or are from grains which failed to twin; only the dislocation substructure is visible. The deformations quoted in the captions are nominal. In very fine-grained polycrystalline material, such as that used here, the structure varies widely from grain to grain after light deformation. More or less average structures are shown. At the higher deformations the structure is more uniform, and the quoted values of deformation probably describe conditions in the individual grains.

The dislocations produced during the initial stages of deformation were generally straight but not smooth as is shown in Fig. 4. Here the

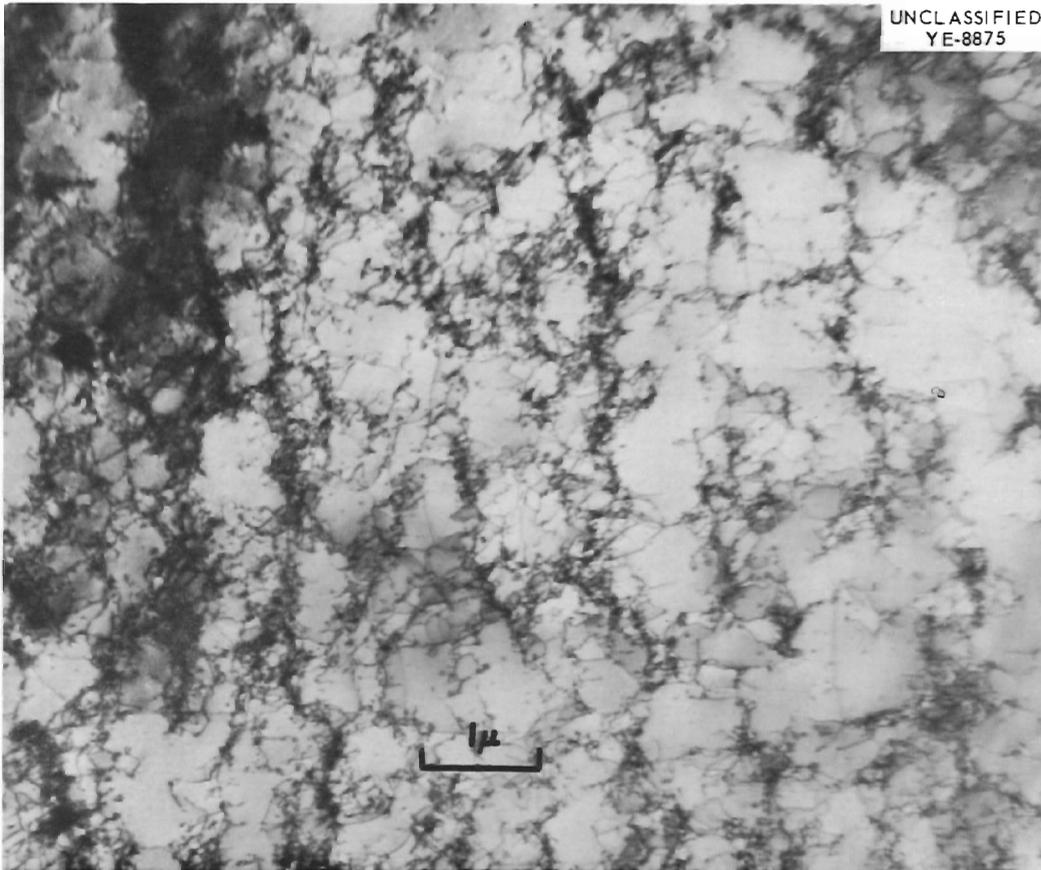


Fig. 3. Dislocation Cell Structure Developed in Pure Columbium Single Crystal by Cold Rolling to 10% Reduction in Thickness at Room Temperature. 16,000X.

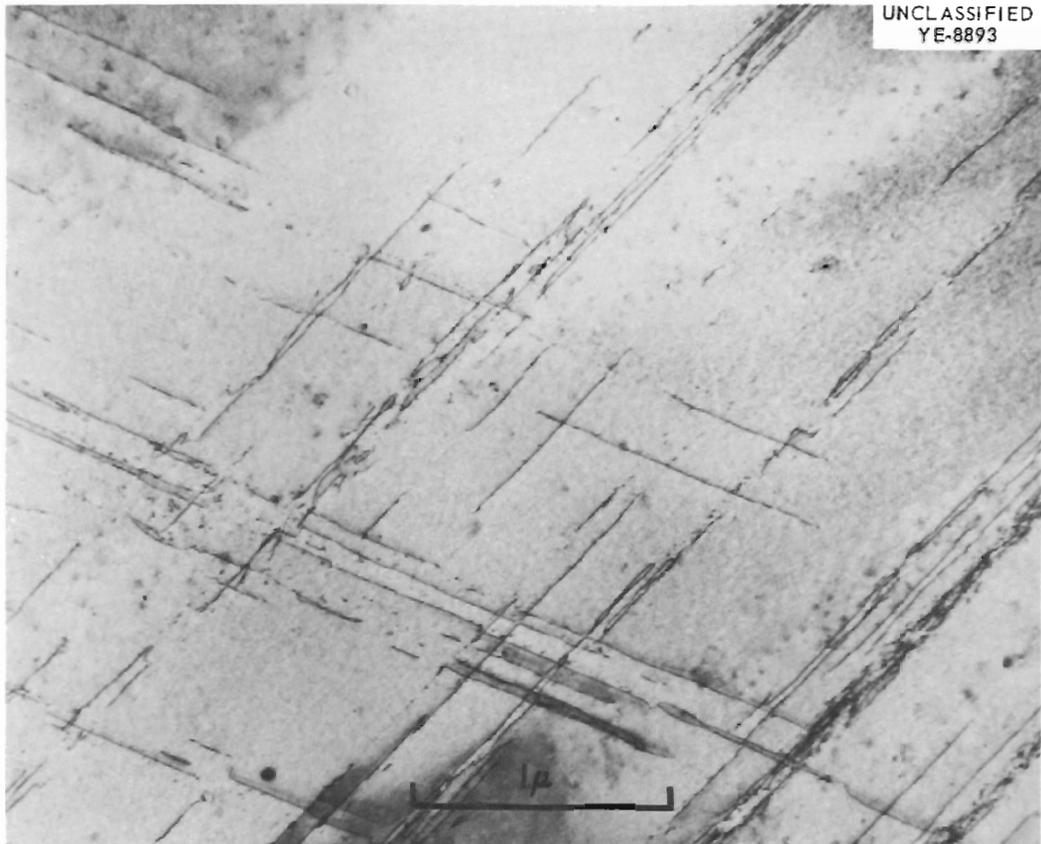


Fig. 4. Dislocation Distribution in Lightly Deformed Cb-40 wt % V. The plane of the foil is (110) and the dislocations, which run along $\langle 111 \rangle$ type directions, are probably not gliding in the foil plane. 35,000X.

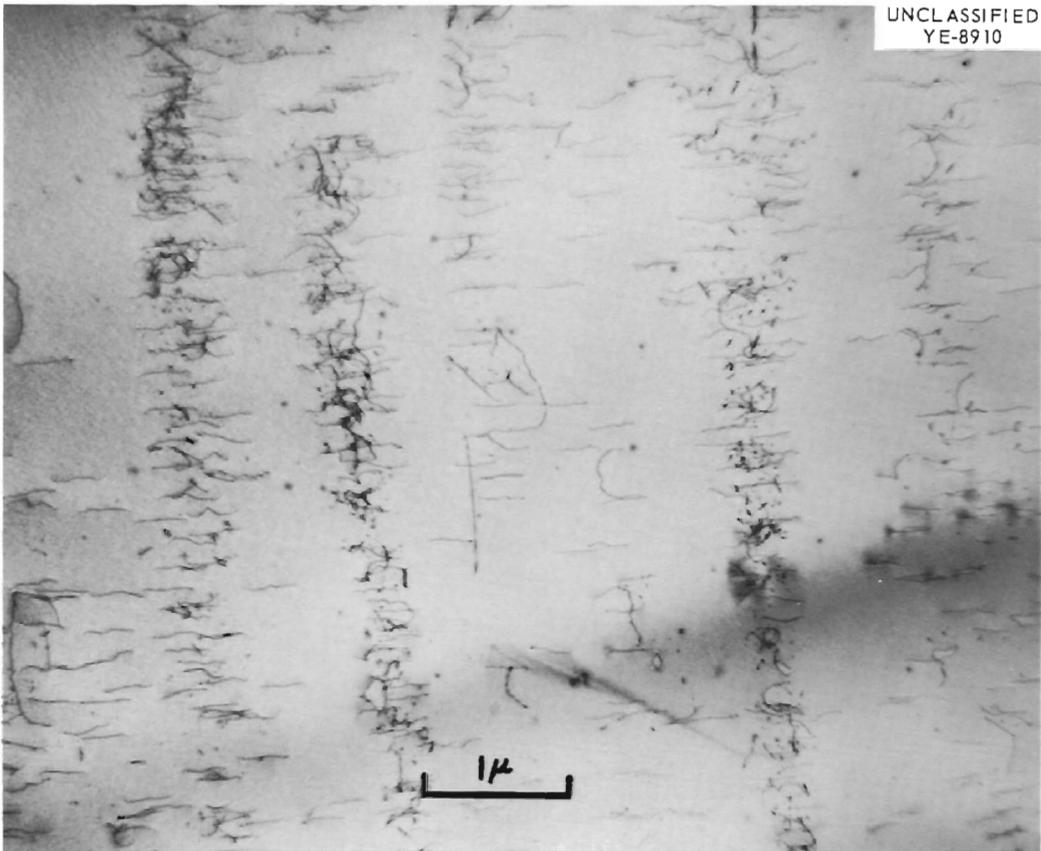


Fig. 5. Lightly Deformed Cb-40 wt % V Foil Showing the Banded Nature of the Dislocation Distribution. 20,000X.

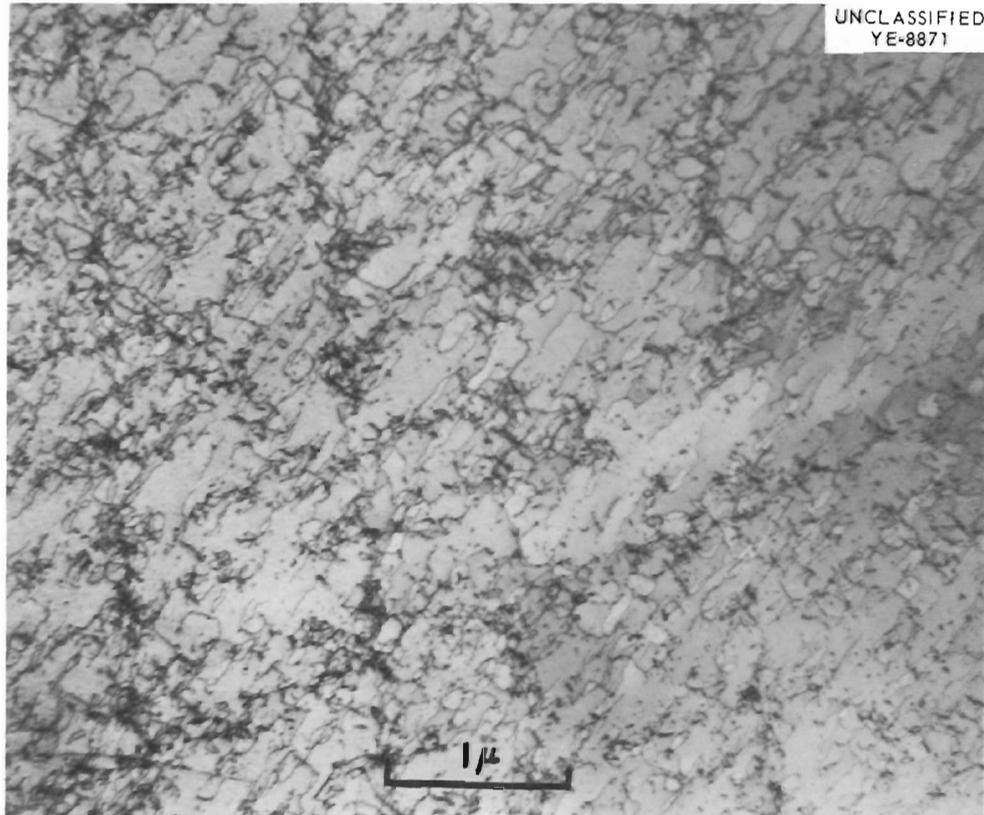


Fig. 6. Dislocation Distribution in Cb-40 wt % V Developed After About 10% Reduction by Rolling. 25,000X.

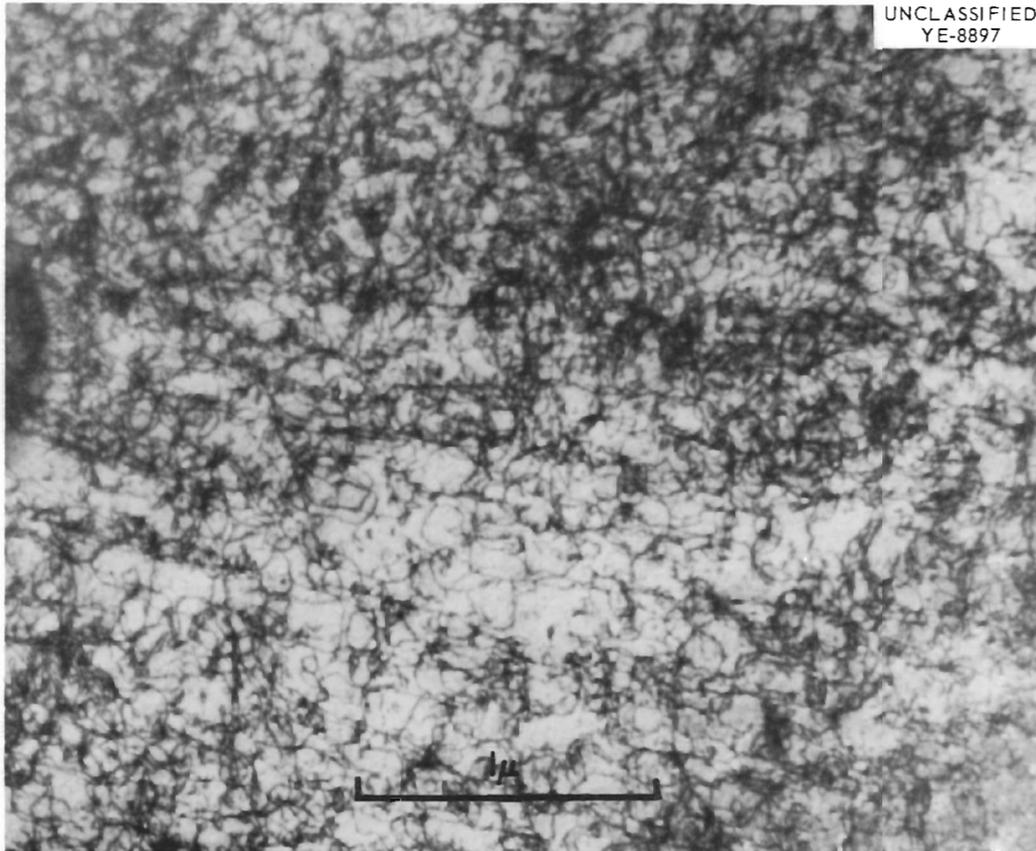


Fig. 7. Uniform Dislocation Distribution in Cb-40 wt % V Developed After 15 to 20% Reduction in Thickness by Rolling. 40,000X.

plane of the foil is (110) and the dislocations lying in the plane run along $\langle 111 \rangle$ type directions, which indicate that they are nearly pure screw. They were probably not moving in the plane of the foil but rather in two of the other {110} type planes inclined at 60° to the plane of the foil. At this stage in the deformation, the slip dislocations appeared to have been confined to bands or discs in three dimensions running roughly parallel to the active slip planes. As the amount of slip in an individual band increased, the dislocations became fairly heavily jogged and a high density of small but frequently elongated loops formed within the band. Tangles were never observed, even at the intersection of two slip systems.

The banded nature of the dislocation structure is better illustrated in Fig. 5 in which the slip plane makes a small angle with the plane of the foil. As was suggested by Fig. 4, the initial dislocations in a band were widely although relatively uniformly spaced. As the dislocation density within the band increased, the dislocations acquired more irregular shapes and numerous loops were formed. The bands or discs were actually warped but of quite uniform thickness nevertheless.

The very straight nature of the initial slip dislocations indicated that the lattice frictional stress must be greatly increased by the alloying. After considerable slip had occurred, the dislocations became heavily jogged so that the different parts of a single dislocation line could not move at the same speed. This in turn gave rise to the more irregular shapes of these dislocations.

After about 10% reduction in thickness by cold rolling, all the dislocations became irregularly shaped and heavily jogged as is shown in Fig. 6, which should be compared with Fig. 3 taken of pure columbium after approximately the same deformation. The density of loops of the specimen was abnormally high - higher than any instance with which these authors are familiar. No tangles were observed in this specimen except at the intersections of the dislocation bands (Fig. 6).

After further deformation the very uniform distribution of dislocations pictured in Fig. 7 resulted. No tangles or configuration even faintly resembling the cell structure were observed at deformations of up to 30%. Pileups were never observed in areas in which only slip had occurred.

The features observed in the 40 wt % V alloy (e.g., straight dislocations, many loops) are similar to those observed by deformation of pure body-centered cubic metals at low temperatures¹² where twinning also occurs, as is illustrated in Fig. 8 for pure columbium. This suggests that the effects produced by solid solution alloying are similar to those of lowered temperature of deformation, both of which tend to promote mechanical twinning. Observations on the Cb-20 wt % V alloy deformed at various temperatures indicated that such an association does indeed exist.

As was noted previously, the twinning transition temperature increases with increasing vanadium content up to 40%, and for the 20% alloy the transition temperature is slightly below room temperature. Specimens of such an alloy deformed by rolling at room temperature were characterized by the presence of the cell structure and the absence of twins, and those by rolling at liquid-nitrogen temperature by dislocation bands and a large number of mechanical twins. The separation of the two structures was almost complete although a few isolated, very thin twins were observed in specimens rolled at room temperature, probably as a result of stress concentrations during rolling.

Depending on the composition of the alloy, individual dislocations responded differently to the stresses generated by the electron beam during examination in the electron microscope. In pure columbium, the dislocations moved so readily that it always was necessary to work at low levels of illumination to keep from altering the structure. The dislocations traveled along highly curved paths typical of other pure body-centered cubic metals.¹² Apparently these dislocations were not at all extended and were, therefore, free to cross slip and follow the highly irregular stress system resulting from the electron illumination. It was generally impossible to move the dislocations in the Cb-40 wt % V alloy even by turning the beam intensity to maximum and removing the condenser aperture. In one instance in a very thin part of a foil, a few dislocations were moved but along a completely straight path. In the 20 wt % V alloy, the ability to move the dislocations lay somewhere between these extremes, and the dislocations traveled along more gently curving paths than in the pure columbium.

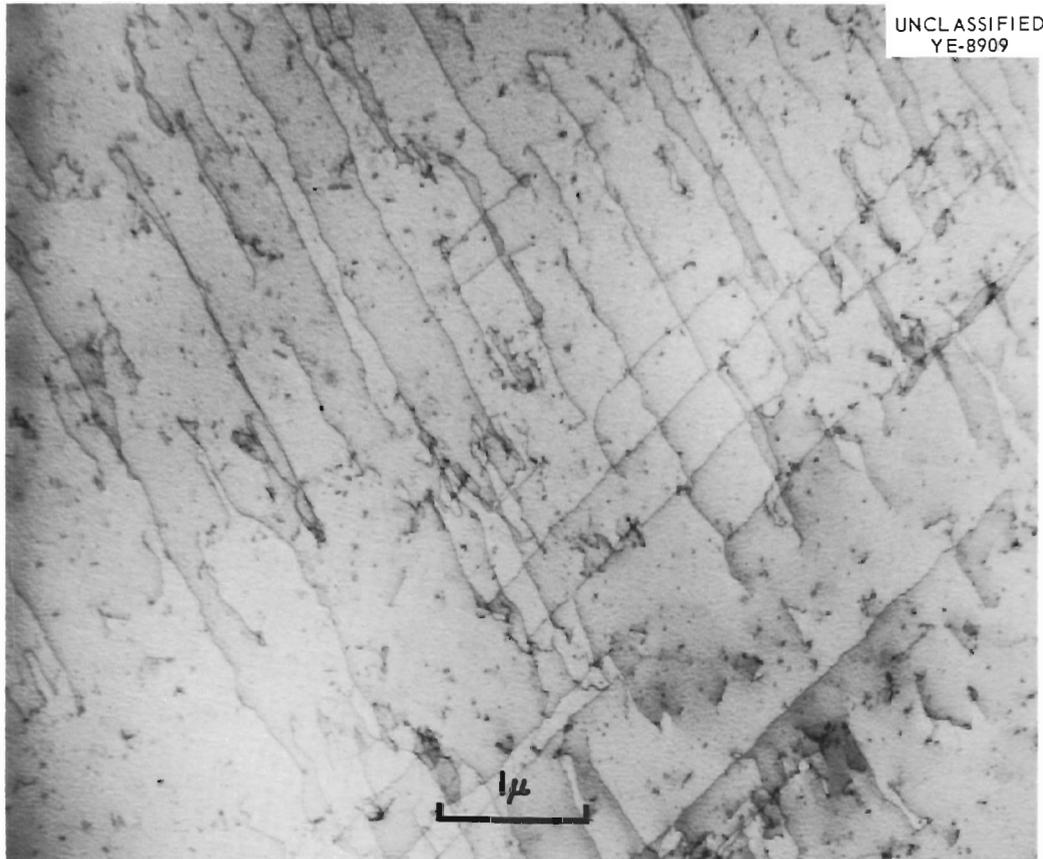


Fig. 8. Dislocation Distribution in Polycrystalline Columbium Pulled in Tension at Liquid-Nitrogen Temperature. The plane of the foil is (110). Compare with Fig. 4. 20,000X.

These results, while only qualitative, show that the addition of vanadium to columbium affects dislocation behavior in two ways. First, it tends to make cross slip more difficult than in pure columbium. Second, it increases the lattice frictional stress on the dislocations to a very high value in the 40 wt % V alloy as is indicated by the immobile, straight dislocations. The decreased cross slip and increased lattice frictional stress produced by alloying are identical with the effect of lowering the temperature of deformation,¹² and in both cases profuse twinning and similar dislocation substructures result. The mechanisms by which solid solution alloying can lead to the unusual twinning behavior will be discussed in the following sections.

Mechanisms of Enhanced Twinning Behavior

Stacking Fault Energy

Intuitively it would appear attractive to attribute enhanced twinning behavior in the columbium-vanadium alloys to a lowering of the stacking fault energy by the alloying action. A lowered interfacial energy ought to reduce the energy of a critical twin nucleus,⁷ and widely extended dislocations separated by the applied stress could serve as twin nuclei. Because of the importance of stacking fault energy on the twinning process, a critical evaluation of the substructure in terms of stacking fault behavior was made.

The severely cold-worked foils of the Cb-40 wt % V alloy were observed to polygonize readily on annealing at moderate to high temperatures. This behavior is not consistent with the hypothesis that the dislocations are extended for the large-scale dislocation rearrangements which are required certainly involve dislocation climb, the activation energy for which would increase if the dislocations were extended. This ought to be especially difficult for the alloys since a cell structure that could serve as a nucleus for the polygonized structure is not present in the deformed material. The ready polygonization of the columbium-vanadium alloys is similar to that observed by Votava¹³ for the Mo-35 at. % Re alloy and suggests that little if any dislocation extension exists in either of these alloy systems.

Regular hexagonal networks were observed in the polygonized sub-boundaries, but in no case was any evidence of alternate extended and contracted node behavior detected. This indicates that the stacking fault energy must be greater than some minimum value of 25-50 erg/cm² which is still moderately high.

This is not, however, a sufficient test of stacking fault energy for the node structure is probably characteristic of the elevated annealing temperature where interstitial contamination undoubtedly occurred and may have pinned the dislocations, preventing node extension. In addition, if stacking fault energy is to be regarded as the dominant factor in determining twinning behavior, the observation that twinning is enhanced at lower temperatures requires that the stacking fault energy decrease as temperature decreases, a rather unattractive thought.

As illustrated by Figs. 4-7, hexagonal networks in which extended node formation could be observed were not formed by room-temperature deformation of bulk specimens. In a few instances, however, in which thinned foils were accidentally damaged during mounting for examination, some regular hexagonal networks were formed. Nevertheless, no extended node behavior was evident, which indicates that the intrinsic stacking fault energy of these alloys at room temperature is moderately high.

The dislocation substructure itself is at variance with the concept of a low stacking fault energy. For example, in face-centered cubic metals of moderate to high stacking fault energy (e.g., nickel) the early stages of deformation are accompanied by the presence of a small number of often elongated dislocation loops. Slight alloying additions (e.g., ~ 5 wt % Al in nickel) that lower the stacking fault energy also eliminate all the loops.¹⁴ The loops probably arise from a cross slip mechanism,¹¹ and the slight extension of the slip dislocations raises the activation energy for cross slip which suppresses the loop formation. Assuming that similar considerations hold for the body-centered cubic system, it is not likely that the stacking fault energy is lowered by the addition of vanadium to columbium as loop production in the 40 wt % V alloy is considerably enhanced over that of pure columbium.

Similar behavior was observed in pure iron deformed in different temperature regions by Keh and Weissmann.¹² In this case, loop formation was enhanced by lowering the temperature of deformation. Keh and Weissmann¹² also observed that the loop size appeared to decrease as the temperature of deformation decreased. No such decrease in loop size was evident as the solute concentration increased in the results described above. Nevertheless, one may make the general statement that alloying affects dislocation loop formation in much the same way as lowered temperature of deformation does.

In face-centered cubic metals of low stacking fault energy the dislocations are both straight and smooth. Although the dislocations in the columbium-vanadium alloys were unusually straight, they were not at all smooth, due undoubtedly to the high density of jogs on them. This further indicates that the dislocations in the alloys are not extended.

Two other observations also suggested that the stacking fault energy is not lowered appreciably. First, no widely extended dislocations separated by a stacking fault ribbon were observed in any of the lightly deformed specimens as frequently occurs in stainless steel, a face-centered cubic alloy of relatively low stacking fault energy. Second, no zig-zagged dislocations¹⁵ were observed in very thin foils due to the equilibrium splitting of partials as also frequently occurs in stainless steel.

The lack of pileups is probably not so much a result of high stacking fault energy as of the nature of the operative dislocation sources (see below). The apparent decrease in ability to cross slip does not necessarily require a low stacking fault energy and indeed may be a result of solid solution effects. Some cross slip over short distances undoubtedly occurs, as is evidenced by the very high density of loops.

It seems rather clear, therefore, that the enhanced twinning behavior in the alloys is not the result of a lowered stacking fault energy but rather of completely different solid solution effects. These will be considered below, but first a few general comments on stacking faults in the body-centered cubic system will be made.

At the present time several reports of experimental evidence for the existence of stacking faults in body-centered cubic metals have been made. All unfortunately have occurred under conditions of doubtful purity or thermal treatment. The early results of Fourdeux and Berghezan¹⁶ and Segall¹⁷ are open to question because the purity of their specimens was low. They did attribute their results to the segregation of interstitials to dislocations, which acted to lower the stacking fault energy, but other observations¹⁸ on purer material in which segregation had obviously occurred failed to show such faults. The structures that they reported as stacking faults may have been plate-like precipitates similar to the nitrides observed by Keh and Wriedt¹⁹ in alpha iron. The remaining example is in the work of Nakayama *et al.*²⁰ on tungsten in which faults and extended dislocations in pileups were formed by straining and/or quenching from temperatures above 2000°C. It is difficult to conceive of pileups being formed under these conditions. At temperatures that high, nearly all the interstitials would be in solution and during the quench, when segregation would be expected to occur, considerable climb and breakup of the pileups should have occurred.

In solid solution alloys, considerable emphasis has been placed on segregation of solute atoms to dislocations to lower the fault energy.²¹ However, the polygonization of the Cb-40 wt % V alloy showed that such behavior did not occur, even at 1200°C. Furthermore, no splitting of "enriched" dislocations by the action of a stress was observed. This behavior coupled with the lack of experimental evidence for the existence of stacking faults in pure metals indicates that separation of dislocations does not occur in body-centered cubic metals under conditions of any practical importance.

Solid Solution Effects

The addition of vanadium to columbium affects dislocation behavior in many different ways, all of which may contribute to some extent to the enhanced twinning properties. As was shown above, the principal change results in an increased lattice frictional stress and a reduced ability of dislocations to cross slip in the alloys. These features in turn radically alter the dislocation substructure which changes the internal stress pattern.

Although the dislocation cell structure has now been observed in most pure face-centered as well as body-centered cubic metals, its origin is still obscure. Li²² has suggested that the tangled cell walls produce a long-range stress field which counteracts the applied stress. Work hardening is a natural consequence since the effective force on a moving dislocation is the difference between the applied stress and the back stress generated by the dislocation tangles. The net stress at arbitrary points within the crystal in general is less than the applied stress. In other words, the dislocation cell structure has the effect of lowering rather than concentrating the internal stresses.

Now the increased frictional stress and retarded ability to cross slip, either singly or together, act to prevent a regrouping of the slip dislocations into the cell structure but instead confine them to the slip bands where stress multiplication rather than reduction probably occurs.

Nothing is known about the signs of the dislocations in a given band or in adjacent bands. The structure of the bands suggests that most of the dislocations in a band are of the same sign although adjacent bands may be of different signs. This is shown in Fig. 5 in which the dislocation density in adjacent bands is often highest on opposite sides of the micrograph. Confining the slip to these narrow bands while requiring the grain boundary to maintain a smooth nonstepped shape results in a complex stress state occurring at the interior of the grain. This can often be relieved by nucleation and growth of a twin between a pair of the bands. This has been observed as is illustrated in Fig. 9.

In addition, the interaction of the dislocations with a grain boundary probably generates high stresses in the adjacent grain which may be relieved by nucleation of one or more twins in the grain. The individual bands are, of course, not pileups but, if they are mostly of the same sign, they introduce stresses in adjacent grains much like those of a pileup. It has been frequently observed in this laboratory, as well as elsewhere,²³ that a series of dislocations approaching a grain boundary will not move into the boundary. One or two actually enter the boundary but the remaining ones either halt or cross slip and enter the boundary at another point,

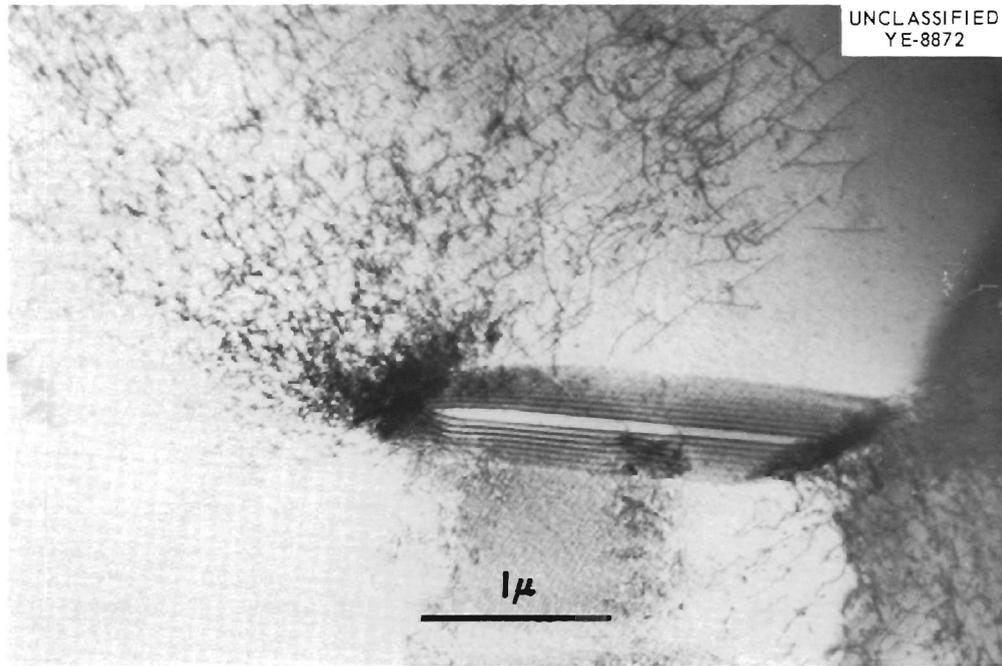


Fig. 9. Short Deformation Twin Nucleated Between Slip Bands in Cold-Rolled Cb-40 wt % V. Note the complex dislocation tangles at the ends. See text for explanation. 25,000X.

apparently from the back stress generated by the microstep formed in the grain boundary. In the high vanadium alloys, long-range cross slip cannot occur readily, and it is possible that the stress can be relieved most easily by nucleation of a twin in the adjacent grain.

No cracking has been observed in the columbium-vanadium alloys after deformation resulting in profuse twinning. If the mechanisms for the formation of cracks either at twin intersections²⁴ or at intersections of trains of emissary dislocations²⁵ have any meaning, many potential crack nuclei were present in these specimens. The absence of cracks indicates that the cleavage strength is raised appreciably by the alloying action.

The alloys also appear to exhibit a greater capacity than pure columbium for holding interstitials in solution. This is demonstrated by the lack of precipitates on grown-in dislocations in the Cb-40 wt % V alloy containing greater than 250 ppm interstitials total. Visible precipitates often are observed¹⁸ after similar treatments of columbium specimens containing less than 150 ppm interstitials, mostly oxygen introduced during recrystallization of cold-rolled foils. Interstitial atmospheres, however, may surround the grown-in dislocations in Cb-40 wt % V.

Similar mechanical behavior has been observed in the Group VI-A metals, tungsten and molybdenum alloyed with rhenium.⁴ The observed ductility is postulated to be a result of lowered interstitial solubility through electronic effects.³ Lattice solubility of interstitials goes to zero in these materials when the electron-to-atom ratio reaches 6. In the pure Group VI-A elements, this ratio in the regions near dislocations and grain boundaries is less than 6, owing to a lack of proper coordination, which results in interstitial atmospheres about grain boundaries and dislocations. The addition of the higher valence element, in this case rhenium, raises the ratio everywhere above 6 and removes all interstitials from solution. This does not account for the twinning behavior but does explain the lack of brittleness in the alloys.

The similar behavior in the columbium-vanadium system cannot be explained in terms of this model, for columbium and vanadium have the same valence, so that the electron-to-atom ratio does not change by

alloying. In addition, the Cb-40 wt % V alloy has a greater solubility of interstitials than pure columbium; no precipitates have ever been observed. Strictly speaking this analogy is not legitimate since pure columbium is very ductile while molybdenum and tungsten are both brittle. Nevertheless, columbium-vanadium alloys containing less than 20 wt % V behave much like dilute molybdenum-rhenium alloys; both are brittle at room temperature. The ductility and enhanced twinning behavior in the higher vanadium content alloys is a solid solution effect of which increased interstitial solubility is one manifestation. Since electronic effects cannot be responsible in the columbium-vanadium case, lattice dilation in the solid solutions probably accounts for the increased solubility.

An additional effect of adding vanadium to columbium is an appreciable raising of the yield strength. When deformation ultimately begins, the general stress level is higher than in pure columbium, which allows through appropriate multiplication the production of local stresses on the order of those required for twinning.

Effect of Interstitials

The observations that the presence of interstitials inhibits the formation and growth of twins have been puzzling for a long time. The normal arguments which one would make generally suggest that interstitials should promote twinning. For example, segregation or precipitation of interstitials at dislocations ought to raise the yield strength and provide sites of stress concentration, both of which should aid in twin formation.

There, unfortunately, is little evidence of the mechanism by which interstitials affect dislocation behavior and interaction. After slow cooling to room temperature, however, interstitial atmospheres or precipitates are present on the grown-in dislocation network¹⁸ even when the concentration of interstitials is below the reported solubility limits.³ This is due to strong binding of interstitials to dislocations through lattice dilation or chemical effects. The grown-in dislocations then are immobilized and consequently play only a passive part in the deformation process. Through this, however, they may be very influential in determining the deformation substructure.

There is experimental evidence that, in columbium²⁶ and molybdenum,²⁷ precipitates on immobile grown-in dislocations serve as anchoring points about which the slip dislocations cluster and tangle. These tangles evidently serve as the nucleus for the cell structure. In columbium in which no precipitates were visible on dislocations, clearly defined cells were not formed by tensile deformation of 10% at room temperature.²⁶ No twins were observed, however, for the specimens were relatively impure and evidently frequent cross slip was induced, either thermally or by submicroscopic clusters of interstitials, which broke up prospective centers of stress concentration.

Interstitials, therefore, may act to prevent twinning by providing sites at which dislocation tangles form or by inducing cross slip, both of which tend to break up the stress concentrations that are necessary for twinning. These sites would be less effective as the temperature is reduced and the concomitant increased lattice frictional stress and decreased ability to cross slip become dominant. The increased tolerance of the substitutional solid solution lattice for interstitials is very important in extending mechanical twinning as a dominant mode of deformation to elevated temperatures.

Structural Features of Twins

In lightly deformed foils, the twins appeared as narrow bands on the order of 1μ wide that generally ran entirely across a grain. When viewed in suitable diffraction conditions, they contained the characteristic fringe structure in the twin matrix interface. That these features were deformation twins was verified by selected area diffraction patterns taken across the twin boundary. Analyses showed that the twin was rotated 180° from the matrix about $[211]$ which is one of several equivalent rotations describing the body-centered cubic deformation twin.

The rare twins which ended within a grain usually contained intense dislocation tangles at their ends, as is shown at both ends of the short twin pictured in Fig. 9. In these instances relatively straight dislocations which were originally part of slip bands were detected beyond the edges of the tangles. The tangles evidently were formed by the interaction

of emissary dislocations²⁸ produced by decomposition of the twinning partials with the band of slip dislocations.

Slip dislocations or groups of slip dislocations apparently cannot be as readily incorporated²⁹ into twins as has been previously thought. The twinning partials are halted as a group at the slip band where the stresses cause them to decompose, probably according to the reaction given by Sleeswyk²⁸ into $\frac{a}{2}$ [111] slip dislocations on every third plane, leaving behind stable groups of $2 \frac{a}{6}$ [111] twinning partials and $1 \frac{a}{3}$ [111] complementary twinning partial. The slip dislocations move out and interact with the dislocations in the slip band to produce the tangles. This reaction then leaves the twin with a blunt rather than a lenticular end.

In some specimens subjected to light bending, a few twins were observed that ended within grains but which had not been stopped by dislocation bands. In these cases, regular arrays of emissary dislocations superficially resembling pileups preceded the twins similar to those described by Votava and Sleeswyk³⁰ for the Mo-35 at. % Re alloy. The spacing of dislocations within the arrays was erratic, probably due to relief of stresses or rearrangement of dislocations during thinning. These trains of dislocations, which were generally several microns long, ended abruptly with no observable obstacles at their tips. The spacing between dislocations usually increased near the end of the train. The twins which produced these arrays were quite thin and all had blunt ends.

A prominent structural feature of twins which has repeatedly been observed by optical microscopy is the notch-like markings on the sides of twins. Hull²⁴ argued that this was a result of slip at the surface when growth of the twin was retarded as it approached a free surface. McHargue,³¹ however, showed that these markings often extended through the crystal and suggested that they were a result of dislocation pileups or other obstacles which acted as barriers to growth.

Transmission electron microscope studies suggest that twins initially form in nearly dislocation-free regions and that they grow in thickness until the stress which nucleated them has been relieved. At this stage the twins are nearly perfect and generally extend entirely across the grains. Notches and serrated markings then develop as slip occurs.

Sleeswyk and Verbraak²⁹ have discussed in detail the dislocation reactions that occur when slip dislocations intersect a coherent twin boundary. In brief, a step is produced at the interface, bounded either by a twinning or by a complementary twinning partial, depending on the crystallographic relationship between the twin and the slip dislocation. If the Burgers vector of the dislocation lies in the twin plane, the dislocation can glide from the matrix to the twin without leaving a step. In the case that a step is formed, the twin can thicken under continued application of stress by motion of the twinning partials and thin by motion of the complementary twinning partials.

Examples have been obtained in which notches have been produced by slip dislocations generated both inside and outside the twin proper as is shown in Figs. 10 and 11.

In Fig. 10 segments of a twin are shown which have been isolated from each other and offset by trains of dislocations from an external source. By tilting the foil a dislocation structure similar to that shown in Fig. 6 was observed in the areas outside the twin. The cutting action, therefore, was not due to trains of slip dislocations of the type illustrated in Fig. 5. The wide spacing between the cuts is in agreement with this interpretation and suggests that emissary dislocations from some short twins in another part of the grain were responsible. The dislocation structure is not what one would expect, based on the models described by Sleeswyk and Verbraak²⁹ where the break should be bounded by a coherent boundary on one side and a stacked-up array of complementary twinning partials on the other. On the opposite side of the twin where the dislocations pass through the twin-matrix interface, these features should be reversed. It is likely that slip dislocations interacted with the complementary twinning partials after the emissary dislocations had passed through. This could produce the complex tangles left at the break.

Similar cutting or notching could be produced by the parallel bands of slip dislocations of the type shown in Fig. 5. This, however, would result in a more uniform notching along the entire length of the twin.

Slip generated by sources inside the twin would produce a similar effect, for when the dislocations pass out through a coherent boundary they may leave a step bounded by a partial dislocation. Such a structure

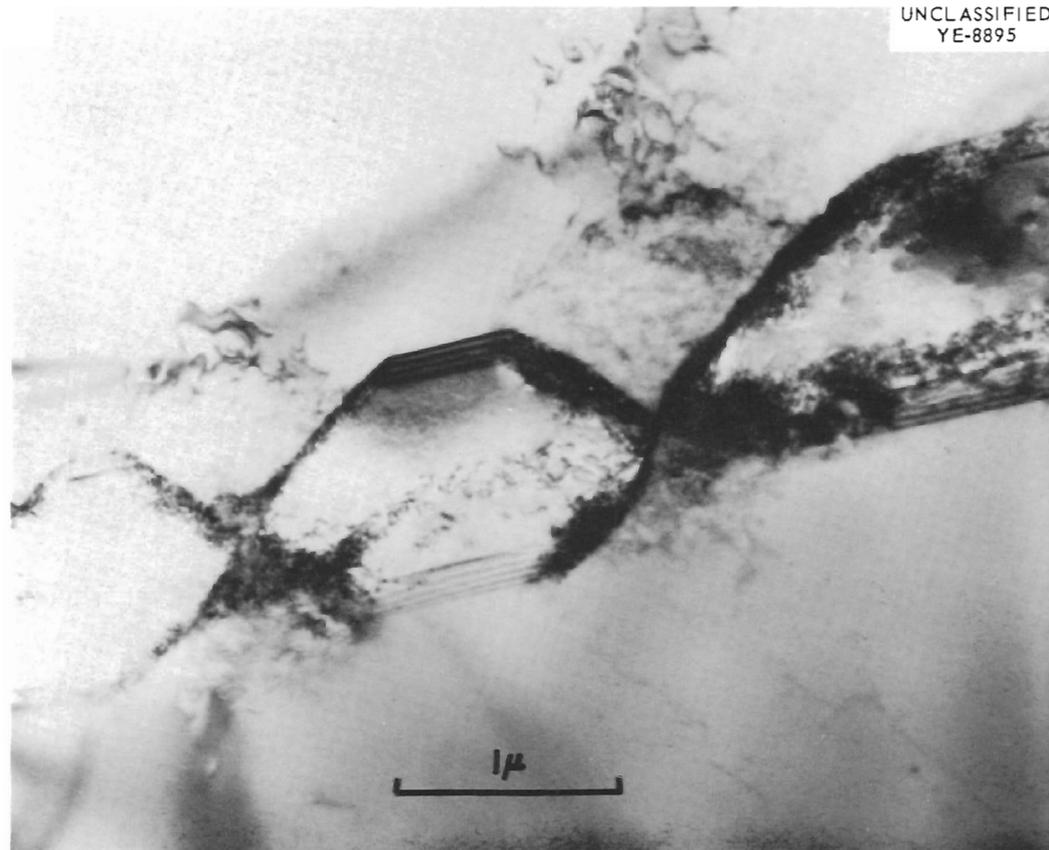


Fig. 10. Notches in a Twin Produced by Cutting Action of Emissary Dislocations. 30,000X.

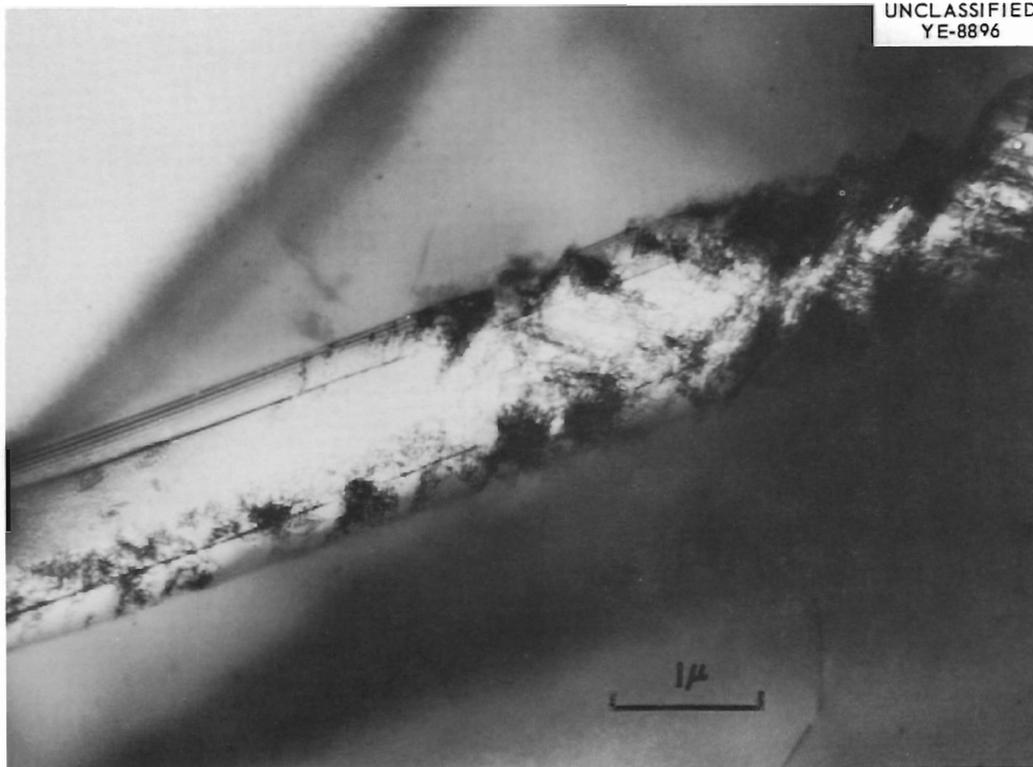


Fig. 11. Notches in a Twin Produced by Dislocations Produced Inside the Twin Cutting Through a Coherent Boundary. 20,000X.

is pictured in Fig. 10. Careful tilting of this specimen failed to reveal bands of dislocations entering the twin where the notches were beginning to form. The dislocation pattern outside the notches was complex, probably due to interaction of the dislocations generated within the twin with the slip dislocations outside it. This type of reaction may be the more common one, for twins are often marked by deep slip steps at their surfaces long before visible slip lines appear in the surrounding material.

Many of the twins contain irregular tangles of dislocations in their coherent boundaries. This structure probably arises from slip dislocations halted at the boundary because the shear stress in the twin is not of suitable magnitude or direction to allow them to glide in the twin.

Some very thin twins of the type described by Hull³² for the molybdenum-rhenium system also have been observed in the Cb-40 wt % V alloy, as is shown in Fig. 12. These twins are only a few atomic layers thick, and the unusual fringe pattern arises from pileups of partials on adjacent slip planes as described by Hull.³² These twins are uncommon and have been produced only in heavily twinned foils which have been roughly handled after being thinned. Critical tilting of the foils containing these twins has failed to reveal the presence of a pole about which the twinning partials could be climbing.

As was discussed above, the twins are believed to be generated by stress concentrations in the crystal. The lack of a pole in the case of the thin twins suggests that a suitable stress is sufficient to nucleate a twin, as was observed by Price⁸ in zinc. There are in these polycrystalline specimens several different mechanisms by which suitable stress concentrations can be produced. The slip bands themselves are capable, either by setting up stress couples within a grain (see Fig. 9) or by interacting with a grain boundary, to generate one or more twins in the adjacent grain. Very often the intersection of a twin with a grain boundary will nucleate a twin in the adjacent grain.

Multiplication of Dislocations

The very interesting dislocation pattern which is observed in specimens that twin has some implications as to possible mechanisms of

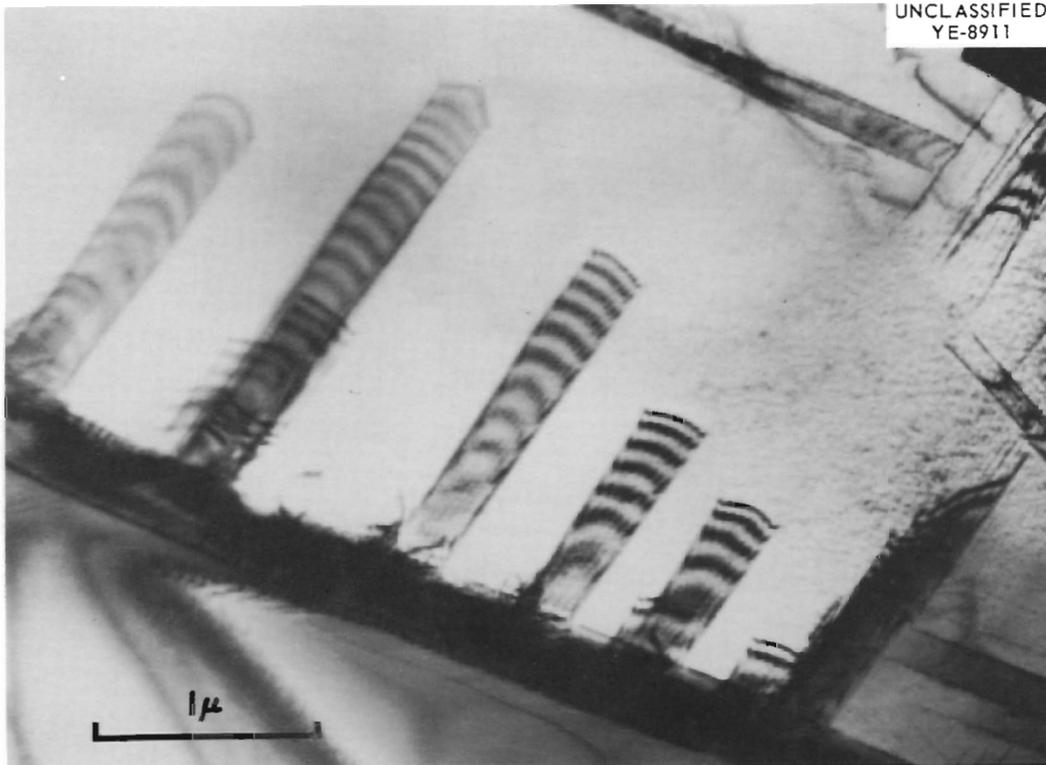


Fig. 12. Thin Twins Produced by Deforming a Prethinned Foil of Cb-40 wt % V. The contrast effects arise from pileups of partials as described by Hull.³² 30,000X.

dislocation multiplication. Because of interstitial pinning, the grown-in dislocations probably do not act as primary Frank-Read generators. Furthermore, the dislocation configuration that is observed is not consistent with any type of generator spinning out large numbers of concentric dislocation loops. If this were the case, pileups frequently would be observed due to the limited ability of the dislocations in the alloy system to cross slip. The only structure resembling a pileup that has been observed is the one of emissary dislocations which precedes some thin twins. The presence of this structure indicates, however, that pileups would be observed if they existed.

Slip is probably transferred from one slip plane to a parallel one a short distance away by relatively infrequent cross slip of dislocation segments due to the high back stress of some barrier, possibly the locked grown-in dislocations or immobile, joggy dislocations on nearby slip planes. The segments which cross slip obviously do not act as secondary Frank-Read sources but rather spread out in the new plane until segments of them come to obstacles and again cross slip. Single ended sources which spin out dislocation spirals could exist, however; but segments of these probably would cross slip before making a complete revolution. A helix of dislocation line, therefore, would be generated. This would lead to regions of the slip bands containing a predominant number of dislocations of one sign.

The active sources probably lie in the grain boundaries. Each source emits several dislocations which move through the lattice before they become heavily jogged and stop. These dislocations then serve as obstacles for later dislocations or, segments of them may break free and multiply, as described above. The smooth, nonjogged nature of the emissary dislocations suggests that the mean free path for dislocation motion in the clean lattice is several microns and that the jogs are not produced by point defects in the lattice. The slip band initially consists of several widely spaced, heavily jogged dislocations. The band then gradually fills up as segments of these multiply and additional fresh dislocations enter from the boundary. At higher deformations, the

bands become more closely spaced and more lateral expansion of the bands occurs to fill up the material with the uniform distribution of heavily jogged dislocations.

Production of Dislocation Loops

Since loops are not observed during the early stages of deformation when the individual dislocations are widely separated and relatively free of jogs, it is likely that they are formed later in the deformation process by large jogs in moving screw dislocations produced by interaction with closely spaced dislocations. That is, as the density of dislocations in a slip band increases, the mutual interaction of closely spaced dislocations leads to more frequent cross slip with resultant jog and loop formation.

Although ease of cross slip is suppressed in the columbium-vanadium alloys over that in pure columbium, it is by no means halted entirely as is evidenced by the high density of loops that is observed. There are two factors that can account for this. First of all, confinement of the dislocations to the slip bands raises the probability of interaction of dislocations that could lead to cross slip. Although cross slip is more difficult in the alloy, this increased frequency of dislocation interaction could lead to a fairly high incidence of cross slip. Second, when cross slip occurs, it appears to do so over short distances. If the distance of cross slip is very large, the two separated dislocation segments can act independently as single ended sources, and no loops will be produced. Low and Turkalo³³ estimate the separation to give this action in silicon-iron crystals deformed at room temperature is about 200 A. Therefore, frequent cross slip over distances less than a few hundred angstroms must occur in the columbium-vanadium alloys, while in pure columbium cross slip generally occurs over much greater distances. Similar observations were made by Keh and Weissmann¹² in iron deformed at low temperatures. They suggested that the increment of cross slip decreased as the temperature decreased due to the increased frictional stress.

Large cusps of the type shown by Low and Turkalo³³ were not present in thin foils of columbium-vanadium alloys. However, their silicon-iron crystals were aged under stress to freeze-in the as-deformed structure. This was not done in these experiments and some relaxation may have occurred during thinning.

CONCLUDING REMARKS

These observations show that dislocation behavior and resultant configurations are significantly altered by solid solution alloying of body-centered cubic metals. The solid solution effects are very similar to the effects produced by lowered temperature of deformation in that the lattice frictional stress is increased and the ability to cross slip is decreased. These effects are accompanied by profuse mechanical twinning in both cases because the dislocation arrangements that are produced tend to concentrate stresses to levels sufficient to nucleate twins. The alloying effect does not lower the stacking fault energy. In addition to raising locally the stress level, the alloying probably raises the cleavage strength and the tolerance of the lattice to hold interstitials in solution. The increased yield strength also raises the general stress level in the crystal. Interstitial precipitates or clusters are believed to inhibit twinning by providing sites at which tangles can occur or by inducing cross slip, both of which tend to break up potential centers of stress concentration.

During the initial stages of deformation of the high vanadium content alloys at room temperature or of pure columbium at low temperature, some slip probably occurs in isolated parts of the specimens. The slip dislocations are unable to regroup themselves to relieve the stress but rather act to multiply it. When the stress locally exceeds the critical value, a stable twin forms and grows to relieve the stress. This twin itself may nucleate twins in adjacent grains or may cause additional slip which, in turn, causes twinning nearby. Slip, therefore, is the trigger which initiates the initial twinning. Once a twin has been formed, however, it can nucleate others and spread the deformation throughout the specimen.

The unusual twinning behavior and resultant ductility in the columbium-vanadium alloys are believed to be due solely to the solid solution effects described above. The electronic effects³ postulated to account for the ductility of the molybdenum-rhenium alloy can have no meaning in this system. The effects in this paper do apply to Group V-A metals but they are of a general nature and suggest that similar effects should occur in Group VI-A. If this is the case, it may be possible to produce alloys of all the refractory metals which have superior strength and corrosion resistance, for example, and at the same time are more workable as a result of being able to twin. Alloying elements which dilate the lattice and do not act as scavengers for interstitial elements should do exactly this.

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