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DISSOCIATION OF $\frac{1}{2}a\langle 111 \rangle$ SCREW DISLOCATIONS IN MOLYBDENUM

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David N. Seidman and J. J. Burke

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DISSOCIATION OF $\frac{1}{2}a\langle 111 \rangle$ SCREW DISLOCATIONS IN MOLYBDENUM*

by

David N. Seidman[†] and J. J. Burke[§]

Cornell University
Bard Hall
Department of Materials Science and Engineering
Ithaca, New York 14850

ABSTRACT

A field ion microscope (FIM) study has been performed of the dislocation structure which exists in the {222} planes and environs of both low purity (approximately $1.2 \cdot 10^{-3}$ at. fr. impurity content) and high purity (approximately $1.7 \cdot 10^{-5}$ at. fr. impurity content) molybdenum specimens. The dislocation structure always consisted of three fold symmetric dissociated $\frac{1}{2}a\langle 111 \rangle$ screw dislocations which emerged normal to the {222} planes. Employing the pulsed field evaporation technique it was shown that the dissociated dislocation structure consisted of a $ka\langle 111 \rangle$ type partial screw dislocation (where k is a constant) which emerged normal to the {222} planes and three stacking faults on the {110} planes that intersected one another along a $\langle 111 \rangle$ type direction. The three stacking faults exhibited three fold symmetry about the $\langle 111 \rangle$ type vector. This represents the first experimental observation of a three fold symmetric dissociated $\frac{1}{2}a\langle 111 \rangle$ screw dislocation in any body centered cubic metal. The dislocation structure was produced in-situ in the FIM specimen at a specimen temperature of less than 50 K as a result of the molybdenum specimens yielding to electric field induced shear stresses which were greater than approximately $4.5 \cdot 10^{10}$ dyne cm^{-2} . It is emphasized that continuous and extensive field evaporation of the specimens never removed this same basic dislocation structure. Simple arguments are presented to show that the observed dissociation could not have been caused by Suzuki segregation effects.

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† John Simon Guggenheim Memorial Foundation Fellow 1972-73.

§ Now at: TRW Metals Division, Minerva, Ohio 44657.

1. INTRODUCTION

The plastic deformation behavior of body-centered cubic (b.c.c.) metals at low temperatures is thought to be controlled by the mobility of screw dislocations (see Hirsch⁽¹⁾ and Christian⁽²⁾ for reviews). Recently there has been a good deal of emphasis on detailed computer calculations of the atom positions in and around the core of $\frac{1}{2}a\langle 111 \rangle$ screw dislocations* (Chang⁽³⁾, Gehlen *et al.*⁽⁴⁾, Vitek *et al.*⁽⁵⁾ and Basinski *et al.*⁽⁶⁾) employing different interatomic potentials that describe stable b.c.c. lattices. The emphasis has been placed on the discrete atomic nature of the dislocation core in b.c.c. metals, because it is believed that the mobility of screw dislocations is determined by an inherent lattice friction stress (the so-called Peierls stress) that is a direct result of the dislocation core structure. The computer calculations indicate that the core structure of $\frac{1}{2}a\langle 111 \rangle$ type screw dislocations in a b.c.c. lattice consists of three intersecting generalized faults on $\{110\}$ planes terminated by fractional dislocations**. It is emphasized that within the context of the computer models the stacking faults are not stable on the $\{110\}$ planes, so that a stacking fault with a constant shear vector ($\vec{R}$) is not formed. For a discussion of the theory of generalized dissociations with unstable stacking faults see Vitek and Kroupa⁽⁸⁾. The important physical result of the computer calculations is that they all show that the $\frac{1}{2}a\langle 111 \rangle$ dislocation cores have three fold symmetry about the $\langle 111 \rangle$

* The quantity a is the lattice parameter of molybdenum.

** A recent summary and discussion of the computer calculations is given by Duesbery *et al.*⁽⁷⁾. The dislocations terminating unstable stacking faults are termed fractional dislocations and should not be confused with partial dislocations. The authors also consider the effect of shear stresses on the dislocation core structure in great detail.

direction. To date there has been no experimental verification of this prediction of the computer calculations, so that the subject has been essentially a theoretical one.

In this paper we present field ion microscope (FIM) observations* of the three fold symmetric dissociation of $\frac{1}{2}a\langle 111 \rangle$ screw dislocations in b.c.c. molybdenum. The molybdenum specimens were deformed at cryogenic temperatures (<50 K) as a result of the high stresses (approximately $4.5 \cdot 10^{10}$ dyne cm^{-2}), produced by the imaging and/or evaporation electric fields. The basic dislocation structure observed was always a three fold symmetric dissociated $\frac{1}{2}a\langle 111 \rangle$ type screw dislocation which emerged normal to the $\{222\}$ planes. Employing the pulsed field evaporation technique it was shown that the dissociated dislocation structure consisted of a $ka\langle 111 \rangle$ type partial screw dislocation (where k is a constant) which emerged normal to the $\{222\}$ planes and three stacking faults on the $\{110\}$ planes that intersected one another along a $\langle 111 \rangle$ type direction. The three stacking faults exhibited three fold symmetry about the $\langle 111 \rangle$ type vector. Each of the three stacking faults was, of course, bounded by a partial dislocation. The experiments were performed on both low purity (approximately $1.2 \cdot 10^{-3}$ at. fr. impurity content) and high purity (approximately $1.7 \cdot 10^{-5}$ at. fr. impurity content) molybdenum, and it is argued that the observed dissociation effects are not caused by Suzuki segregation of impurity atoms to the stacking faults.

* The observations reported in this paper were made during the course of an FIM study(9) of the temperature dependence of the ionization and field evaporation characteristics of molybdenum. This study was also aimed at understanding the contrast effects detected in molybdenum images, so that the contrast effects produced by point defects induced by either irradiation or quenching could be properly interpreted.

2. EXPERIMENTAL TECHNIQUES

2.1 Specimen preparation

The experiments were performed on two different grades of molybdenum. The first grade was Sylvania 0.25 mm diameter polycrystalline wire with a resistivity ratio, $R(R=R_{298\text{ K}}/R_{4.2\text{ K}})$ of 33. The results of a spark source mass spectrometric analysis of this material are shown in Table 1. The sum of all the maximum values of the concentrations listed is approximately $6.5 \cdot 10^{-3}$ at. fr. If it is assumed that an average value for the resistivity of an impurity atom is $1.5 \cdot 10^{-4}$ ohm cm (at.fr.)⁻¹, then the value $R = 33$ implies a total impurity atom concentration of approximately $1.2 \cdot 10^{-3}$ at. fr. Hence, the spectrometric analysis and the residual resistivity measurements at 4.2 K yielded impurity concentrations that were in agreement with one another within a factor of about 5. It is noted that the major impurities in this commercial grade molybdenum were oxygen, nitrogen and iron.

The second type of specimen employed was prepared by zone refining 1 mm diameter rods of Sylvania 99.9 wt. % pure molybdenum. The processing treatment consisted of the following four steps: (1) two pass zone-refining in a background pressure of $5 \cdot 10^{-6}$ Torr; (2) a 6 h anneal at about 2200°C in a background pressure of less than $5 \cdot 10^{-9}$ Torr; (3) an 8 h anneal at approximately 2200°C in a partial pressure of oxygen of $5 \cdot 10^{-5}$ Torr; and (4) an 8 h anneal at 2200°C in a background pressure of less than $2 \cdot 10^{-9}$ Torr. The value of R for this second type of specimen was $2.2 \cdot 10^3$, uncorrected for any possible specimen size effect, as measured on a 1 mm diameter specimen. The residual resistivity at 4.2 K corresponds to an average total impurity concentration of approximately $1.7 \cdot 10^{-5}$ at. fr.

employing an average impurity atom resistivity of $1.5 \cdot 10^{-4}$ ohm cm (at.fr.)⁻¹.

A comparison of Tables 1 and 2 shows that in the $R = 2.2 \cdot 10^3$ specimen the elements cobalt, rubidium, niobium, arsenic, sulfur, copper and potassium were undetected whereas they were detected in the $R = 33$ specimen. Presumably these elements were removed by a combination of the zone refining and annealing treatments described above. It should also be noted that the concentration ranges for both tungsten and silicon were identical for the $R = 33$ and $2.2 \cdot 10^3$ specimens. The segregation coefficient of tungsten in molybdenum is nearly unity⁽¹⁰⁾ and therefore it cannot be removed by zone refining. Since the segregation coefficient of silicon in molybdenum is less than unity⁽¹¹⁾ it should, in principle, be removed by zone refining. Although the range of concentration for silicon has a factor of 10 spread it is certainly possible that some purification occurred, but was undetected because of the poor resolution of the mass spectrometric technique.

The sum of the maximum values of the impurity concentrations listed in Table 2 is approximately $1.6 \cdot 10^{-3}$ at. fr. Hence, the total impurity atom concentration determined by spectrometric analysis differed from the average impurity atom concentration determined by the 4.2 K resistivity by a factor of about 10^2 . This difference[†] is most likely due to the fact that the high temperature

† Eyre, Maher and Bartlett(12) have conducted a more detailed chemical analysis study of zone refined molybdenum than the one performed by us and they found that the R values are a good measure of the impurity atom concentration. Although they, also, found that the total impurity concentration (see Tables 1, 2 and 3 in Eyre *et al.*) as measured chemically exceeds that predicted by the residual resistivity at 4.2 K, if one employs an average impurity atom resistivity of $1.5 \cdot 10^{-4}$ ohm cm (at.fr.)⁻¹. This result lends credence to our argument that some of the purification occurs via the mechanism described in this paragraph.

anneal in the presence of a partial pressure of oxygen caused the formation of metal oxide precipitates which were distributed on a rather coarse scale throughout the specimen. A coarse distribution of metal oxide precipitates will result, of course, in a low value of the 4.2 K resistivity, whereas the mass spectrometric analysis does not distinguish between impurity atoms in solid solution versus ones in precipitates.

2.2 Electropolishing procedure

All the specimens were prepared by the standard drop-off technique⁽¹³⁾ in a volumetric solution of seven parts methanol to one part sulfuric acid at 5 to 15 Vdc. Each specimen was transferred immediately to an FIM after a sharp tip was obtained. The FIM was evacuated to 10^{-5} Torr within less than 1 h from the time of completion of the electropolishing procedure. This rapid transfer technique reduced the number of tips that flashed prematurely. The flashing of tips was attributed to the formation of either a heavy layer of an oxide or a nitride of molybdenum on the tip's surface.

2.3 Experimental apparatus

The experiments were performed in two different types of FIM's. The first type was an all stainless steel diffusion pumped FIM which was baffled with a Granville-Phillips liquid nitrogen cold trap. The pressure in this FIM, prior to backfilling with helium gas was always in the range $(0.7 \text{ to } 2) \cdot 10^{-8}$ Torr. This FIM was used to examine the specimens with $R = 33$. The second type of FIM was the one described previously by Chen and Seidman⁽¹⁴⁾ and Berger et al.⁽¹⁵⁾. The background pressure attained in this FIM prior to backfilling

with helium gas ($2.5 \cdot 10^{-4}$ Torr) was $6 \cdot 10^{-10}$ Torr. This FIM was used to examine the $R = 2.2 \cdot 10^3$ specimen. The FIM liquid helium cryostat, the temperature measurement problem, and photographic recording system have been described by Seidman et al.⁽¹⁶⁾, Seidman and Scanlan⁽¹⁷⁾ and Scanlan et al.⁽¹⁸⁾. The image was intensified with an internal image intensification system based on a 76 mm diameter Bendix channel plate multiplier (Turner et al.⁽¹⁹⁾ and Brenner and McKinney⁽²⁰⁾). The intensified image was recorded on 35 mm ciné film (Kodak Plus-X) with an Automax ciné camera. The shutter of the camera was synchronized with the output of a zero to 5 kV pulse amplifier (Robertson and Seidman⁽²¹⁾), so that the specimen was alternately pulsed for 100 μ sec to remove 1 or 2 atoms from a typical high index plane and then photographed at best image field between the pulses. The 35 mm ciné film was analyzed with a Vanguard motion analyzer⁽¹⁸⁾ which handled the 1000 ft rolls of film employed. The photographic recording and analyzing techniques were similar to those employed in our laboratory for the study of point defects in irradiated or quenched metals (see Seidman⁽²²⁾ for a review of this research).

3. ANALYSIS OF DISLOCATION CONTRAST

In this section we review briefly the analytical concepts necessary to understand the dislocation contrast[†] effects which are produced by perfect dislocations, stacking faults and partial dislocations.

[†] The reader is referred to Bowkett and Smith⁽²⁴⁾, Smith et al.⁽²⁵⁾ and Smith⁽²⁶⁾ for more details concerning the geometric theory of FIM contrast effects from dislocations. In this paper we employ the notation of Smith and co-workers.

3.1 Perfect dislocations

Any perfect dislocation line in a crystal lattice converts a set of initially parallel atomic planes into a helical ramp (Cottrell⁽²³⁾). The Burgers vector and the unit line vector of the perfect dislocation are denoted by $\vec{b}$ and $\hat{l}$ respectively, and the unit normal to a set of rational lattice planes by $\hat{n}$. The above geometric property is valid for all dislocations provided that $\hat{n} \cdot \vec{b}$ and $\hat{n} \cdot \hat{l}$ do not equal zero.

Now consider the case where a dislocation line emerges in a single pole of an FIM surface and the Burgers circuit is drawn in the plane of $\hat{n}$. A circle sketched in the plane of $\hat{n}$ in "good crystal" (Read⁽²⁷⁾) is transformed into one turn of a helix of pitch $\hat{n} \cdot \vec{b}$ (see Fig. 1 in Smith et al.⁽²⁵⁾). The value of $\hat{n} \cdot \vec{b}$ is given by

$$\hat{n} \cdot \vec{b} = p d_{hkl} \quad (1)$$

where

$$p = hu + kv + lw. \quad (2)$$

Since $\vec{b}$ is a lattice vector for a perfect dislocation and $\hat{n}$ is the unit normal to a set of rational planes, the pitch will always be equal to an integral number of interplanar spacings[†] (d_{hkl} 's). If $p = 1$ the contrast effect produced is a spiral^{*} with a pitch equal to one d_{hkl} . In general when p is an integer greater than unity then the pitch of the spiral is $p d_{hkl}$, and the contrast effect

† For a b.c.c. lattice $h+k+l$ must be an even number.

* Pashley(29) and Ranganathan(30) appear to have been the first ones to have realized independently that the helical ramp property of a dislocation would give rise to a spiral type contrast effect in an FIM image. Ranganathan(30), Sanwald and Hren(31), and Brandon and Perry(32) have used the condition that in order for a dislocation to be visible in an FIM image that $\vec{g} \cdot \vec{b}$ (where $\vec{g}$ is the reciprocal lattice vector of the plane involved) be non-zero in analogy with the electron microscope condition for visibility. It is noted that this approach and the approach of Smith and co-workers are equivalent.

is p interleaved spirals (see Smith et al.⁽²⁶⁾ and Sanwald et al.⁽²⁸⁾ for illustrations of p equal to 2 and 3).

3.2 Stacking faults

Consider the case of a planar stacking fault where the displacement at the fault is given by the vector $\vec{R}$, and where the magnitude of $\vec{R}$ is the same everywhere on the fault surface. Denote the unit normal to the fault surface as $\hat{s}$ and exclude the case where $\hat{n} \times \hat{s} = 0$ from the following discussion. The intersection of the stacking fault with a pole of an FIM surface results in a step of height $\hat{n} \cdot \vec{R}$ normal to the surface where

$$\hat{n} \cdot \vec{R} = q d_{hkl} \quad (3)$$

and

$$q = h u_p + k v_p + l w_p \quad (4)$$

since $\vec{R} = [u_p \ v_p \ w_p]$. The quantity q need not be an integer in this case since u_p , v_p and w_p are not necessarily integers. When the value of q is nonintegral and greater than one the step height after field evaporation will be $q' d_{hkl}$, where q' is the nonintegral remainder after an integral number of planes have been removed.

A schematic example of stacking fault contrast is shown in Fig. 1. Figure 1a is a section through a pole of an FIM specimen prior to the introduction of the fault, while Fig. 1b shows the same pole after the introduction of a stacking fault. The case drawn is for $|q| < 1$. The contrast expected in the FIM image is shown in Fig. 1c, after the crosshatched piece in Fig. 1b has been removed by field evaporation. Note that the "lines of contrast"* in Fig. 1c are all

* The line which separates the two halves of the fault and which is parallel to $\hat{n} \times \hat{s}$ is called a "line of contrast".

parallel to the vector $\hat{n}\hat{s}$. For a schematic illustration of the case where $\hat{s}$ is perpendicular to $\hat{n}$ and $1 < |q| < 2$ see Smith et al. (25).

3.3 Partial dislocations

Since a partial dislocation bounds a stacking fault, the contrast effects observed in a pole of an FIM specimen are explicable in terms of the analyses presented in Sections 3.1 and 3.2. For a partial dislocation with a Burgers vector $\vec{b}_p$ a spiral with a pitch equal to $\hat{n} \cdot \vec{b}_p$ will be formed. In terms of d_{hkl} the pitch q is given by equation (3) with $\vec{R} = \vec{b}_p$. If q is nonintegral then a stepped spiral will be the resulting geometric configuration. An example of the expected contrast effect produced by a single partial dislocation bounding a stacking fault and emerging in the center of a pole of an FIM specimen is shown in Fig. 2 for the case $0 < |q| < 1$. If q is integral for a partial dislocation then a kinked spiral is the resulting geometric configuration. A detailed discussion of this effect and further examples for other values of q is given by Bowkett and Smith (24) and Smith et al. (25).

4. RESULTS

4.1 Pertinent crystallographic information

The experimental observations which we will discuss involve the regions of the surface of FIM specimens which are centered on $\{222\}$ planes. The crystallography relevant to this region is summarized by the portion of a standard 222 stereographic projection which is shown in Fig. 3. The $\{hkl\}$ planes involved in the interpretation of the FIM micrographs are the (222) , $\{233\}$, $\{244\}$ and

{011} planes. It is noted that the $(\bar{1}01)$, $(\bar{1}10)$ and $(01\bar{1})$ planes are all perpendicular to the (222) plane and have a [111] zone axis.

An FIM micrograph of a molybdenum specimen, recorded at a specimen temperature (T_T) of 12.7 K, with an R value of $2.2 \cdot 10^3$ and a [111] single crystal orientation is shown in Fig. 4. This micrograph should be compared with the 222 stereographic projection shown in Fig. 3.

4.2 Observations of three fold symmetric dissociation of $\frac{1}{2}a\langle 111 \rangle$ screw dislocations

The main result obtained for all specimens (i.e., both the R equal 33 and $2.2 \cdot 10^3$) examined was that every (222) plane and environs examined showed contrast effects which are characteristic of the three fold symmetric dissociation of $\frac{1}{2}a\langle 111 \rangle$ type screw dislocations. For all the dislocation contrast effects we observed the vector $\hat{i}$ was always of the form $\langle 111 \rangle / \sqrt{3}$; that this is the case will be seen from our explanation of Figs. 5 to 7. In the b.c.c. lattice a total dislocation which lies along a $\langle 111 \rangle$ direction must be a screw dislocation with $\vec{b}$ equal to $\frac{1}{2}a\langle 111 \rangle$.

The first contrast effect we shall discuss is that caused by the stacking faults on {110} type planes. Figure 5, which illustrates this point, is a ten frame pulsed field evaporation sequence, out of a total of 181 frames of ciné film, that exhibits only the (244) and (233) planes. The other $\{244\}$ and $\{233\}$ planes with a [111] zone axis also simultaneously exhibited this contrast effect, but we shall discuss this case first for the purpose of clarity. The line of contrast that runs through the (244) and (233) planes is the trace of the $(01\bar{1})$ plane. Note that between frame numbers 17 and 20 the atoms that are imaged in the (233) plane shift from the right

to the left hand side of the line of contrast. This same contrast effect in the (233) plane repeats itself between the following pairs of frame numbers: 47 and 59; 79 and 105; and 125 and 181. The above contrast effect also occurs in the (244) plane as an examination of the following pairs of frame numbers demonstrates: 20 and 47; 59 and 79; 105 and 114; and 114 and 125 in Fig. 5. The line of contrast seen in Fig. 5 remained in the same position on the FIM pattern as approximately sixty (222) planes were removed by the pulsed field evaporation technique and recorded $4.5 \cdot 10^3$ frames of film. This result implies that the plane of the stacking fault must be an $(01\bar{1})$ plane. Note that if the plane of the stacking fault was inclined to the surface then the line of contrast, which is parallel to the vector $\hat{n} \times \hat{s}$, would have shifted parallel to itself across the (233) and (244) planes as successive atomic planes were removed. This parallel shifting motion of the line of contrast for a stacking fault which is inclined to a surface is illustrated schematically in Fig. 1c.

A more detailed example of this first contrast effect is exhibited in Fig. 6. The presence of three lines of contrast caused by the traces of the three $\{110\}$ planes that are perpendicular to the (222) plane can be seen in this ten frame sequence (out of 224 frames of film). The three lines of contrast caused by the $(01\bar{1})$, $(\bar{1}10)$ and $(\bar{1}01)$ planes in the (233), (332) and (323) planes respectively can be observed simultaneously by inspecting frame numbers 14, 47, 84 and 128. It can also be seen that the imaged atoms in the different (233) planes shift across the line of contrast as successive (222) planes are removed. (A total of sixty (222) planes were

removed and recorded on $4.5 \cdot 10^3$ frames of ciné* film.) For example, note the positions of the imaged atoms shifting from one side of the line of contrast to the other side in the (233) plane between the following pairs of frame numbers: 1 and 14; 26 and 47; 47 and 64; and 154 and 224. It is emphasized that the three different lines of contrast seen in Fig. 6 remain in the same position as successive (222) planes were removed between frame numbers 1 and 224. This means that the three intersecting stacking faults which cause the three different lines of contrast must lie on the {110} planes.

The second contrast effect we wish to discuss is the stepped spiral effect produced by a partial dislocation (see Section 3.3). Examples of the stepped spiral effect in the (222) and {332} planes can be seen in both Figs. 5 and 6. The existence of a stepped spiral appears as a step (or discontinuity) in a ring of atoms in the (233) planes seen in Fig. 5. Some of the steps are indicated by vees in frame numbers 17, 20, 59, 79 and 125 in Fig. 5. The schematic illustration of a stepped spiral in Fig. 2 should help the reader in confirming the presence of these steps (or discontinuities) in the ring structure.

The stepped spiral contrast effect appears in both the (222) and {332} planes in the FIM micrographs exhibited in Fig. 6. The (222) plane is particularly interesting in that three different steps are present in the outermost ring of atoms in this plane. The two vees in frame 1 of Fig. 6 point to two imaged steps in the (222) plane. Another example of this effect is also seen in frame number 84 where the three vees in the (222) plane point to the three steps.

* A 16 mm ciné film showing the pulsed field evaporation through approximately 60Å of metal, and which contain a dissociated $\frac{1}{2}a\langle 111 \rangle$ screw dislocation is available upon request.

Figure 7 is a schematic illustration* of the three steps observed in the (222) plane.

The third aspect of the contrast which we wish to mention is the presence of a line of contrast through the {244}, but the apparent absence of a step or discontinuity in the ring structure through this plane. This effect is particularly noticeable in frame numbers 17, 20, 79, 105, 125 and 181 in Fig. 5.

It is emphasized strongly that we were never able to obtain a perfect crystal in the (222) planes and environs as a result of continuous and extensive field evaporation. Furthermore, elements of the dislocation contrast effects discussed in this section may be seen in all of the single frame micrographs of molybdenum published in the literature (e.g. see Bowkett and Ralph⁽³³⁾ and Müller and Tsong⁽³⁴⁾). Thus it is believed that the deformation of a molybdenum specimen in the stress field caused by the required imaging and/or evaporation electric fields always results in the same basic dislocation structure in the (222) regions of a molybdenum FIM specimen.

4.3 Separation of partial dislocations

The separation between the central partial dislocation which lies parallel to the $\hat{z} = \langle 111 \rangle / \sqrt{3}$ vector and the second partial dislocation which must terminate each of the {110} stacking faults was calculated in the following manner. First, the local radii of curvature were calculated, using the ring counting method, between the (222) plane and the {244} planes, employing the equation⁽³⁵⁾

$$r_{(222)-(244)} = \frac{d_{222}^N}{1 - \cos \theta_{222-244}} = 24NA^\circ \quad (5)$$

* See Fig. 4 in Smith's⁽²⁶⁾ article for an isometric drawing of this contrast effect.

where N is the number of rings between the 222 and 244 poles which are separated by the angle $\theta_{222-244}$. Then the separation between partials (ρ_i) was taken to be given by the maximum radius* of the i^{th} (222) ledge constituting a field-ion pole, and this quantity was calculated from the expression⁽³⁶⁾

$$\rho_i = [2r_{(222)} - \{244\} id_{222} - id_{222}^2]^{1/2} = (2r_{(222)} - \{244\} id_{222})^{1/2}. \quad (6)$$

The results for the specimens exhibited in Figs. 5 and 6 are presented in tabular form in Tables 3 and 4. The uncertainty of $\pm 24\text{\AA}$ in $r_{(222)} - \{244\}$ arose from an assumed uncertainty of ± 1 in the value of N. The uncertainty of $\pm 7\text{\AA}$ in ρ_i was calculated from $d\rho_i/\rho_i$ assuming a value of ± 1 for the quantity di . It is noted that the specimen (Table 4) with the smaller values of $r_{(222)} - \{244\}$ had the smaller values of ρ_i (compare Tables 3 and 4).

Another uncertainty that enters into the measurement of the ρ_i is the determination of the exact position of the second partial dislocation. This was partially taken care of by assuming an uncertainty of ± 1 in the value of N, but we note that this is not an exact treatment of this error.

5. DISCUSSION

5.1 The dissociated three fold symmetric dislocation structure

The dislocation structure observed always consisted of three fold symmetric dissociated screw dislocations which emerged in the {222} planes. This dislocation structure was produced in-situ in the FIM specimens at a T_p of less than 50 K as a result of the molybdenum specimens yielding to the electric-field induced shear

* See Fig. 1.18 in Bowkett and Smith(24).

stresses which were greater than* approximately $4.5 \cdot 10^{10}$ dyne cm^{-2} . The absence of edge dislocations in our deformed molybdenum FIM specimens is consistent with the electron microscope results⁽³⁷⁾ on single crystal molybdenum deformed at less than 0.14 of the melting point. The reason for the presence of only screw dislocations is their low mobility at cryogenic temperatures, whereas the edge dislocations are highly mobile at these same temperatures and presumably have slipped out of the FIM specimens.

The structure observed in the {222} planes consisted of a partial $\langle 111 \rangle$ screw dislocation emerging normal to a {222} type plane with stacking faults lying on the three {110} planes which intersected one another along a $\langle 111 \rangle$ type direction (see Figs. 3 and 6). The three stacking faults intersect one another parallel to a $\langle 111 \rangle$ type direction and have three fold symmetry about this vector. A schematic drawing of this dislocation structure is shown in Fig. 8. The stacking faults which lie on the three {110} planes are indicated by crosshatching and the four partial dislocations involved by the Burgers vectors $\vec{b}_s$, $\vec{b}_1$, $\vec{b}_2$ and $\vec{b}_3$. The dissociation of the perfect $\frac{1}{2}a[111]$ screw dislocation must involve a reaction of the form:

$$\begin{aligned} \frac{1}{2}a[111] + \vec{b}_1 \text{ on the } (\bar{1}01) \text{ plane} \\ + \vec{b}_2 \text{ on the } (\bar{1}\bar{1}0) \text{ plane} \\ + \vec{b}_3 \text{ on the } (01\bar{1}) \text{ plane} \\ + \vec{b}_s \text{ at the intersection.} \end{aligned} \quad (7)$$

The vector $\vec{b}_s$ is of the form $ka[111]$ where k is a constant which

* Calculated from the expression $\frac{1}{2}(E^2/8\pi)$ where E is the evaporation field⁽³⁸⁾ of molybdenum ($4.5 \cdot 10^8$ volt cm^{-1}). This stress is comparable to the shear modulus of molybdenum ($12.8 \cdot 10^{11}$ dyne cm^{-2}) listed by Friedel⁽³⁹⁾.

depends on type of dislocations employed for $\vec{b}_1$, $\vec{b}_2$ and $\vec{b}_3$. Both Kroupa⁽⁴⁰⁾ and Kroupa and Vitek⁽⁴¹⁾ have suggested the following reaction for the three fold symmetric dissociation of a $\frac{1}{2}a[111]$ screw dislocation:

$$\frac{1}{2}a[111] + \frac{1}{8}a[101]_{(\bar{1}01)} + \frac{1}{8}a[110]_{(\bar{1}10)} + \frac{1}{8}a[011]_{(01\bar{1})} + \frac{1}{4}a[111]. \quad (8)$$

Smith⁽²⁶⁾ has analyzed the contrast effects expected from Eqn. 8 for the case where all the partial dislocations and the stacking faults are imaged in one pole of a field-ion microscope specimen. The contrast effects we observed involved a number of poles, and therefore we must discuss our micrographs in more detail. In our case the partial dislocation ($\frac{1}{4}a[111]$) which appears at the intersection of the three stacking fault planes has a q value (see Eqn. 4) of $1\frac{1}{2}$. This implies that we should see a double stepped spiral, which is intersected by three lines of contrast caused by the three $\{110\}$ stacking fault planes. A schematic drawing of the contrast effects we actually observed in the (222) plane is shown in Fig. 7. The presence of these three steps in the only ring of imaged atoms in the (222) plane is readily observed in Fig. 6. The problem here is that we have not been able to observe a second ring of atoms which we could clearly associate with the (222) plane as opposed to belonging to the $\{233\}$ planes. Hence we could not verify the presence of a double stepped spiral contrast effect.

The second point we wish to make involves the absence of a step (see Section 4.2) in any of the rings of atoms imaged in the $\{244\}$ planes. This result is consistent with the fact that q is equal to unity for the three $\{244\}$ planes and the respective $\frac{1}{8}a\langle 110 \rangle$ type Burgers vectors involved, as the contrast theory predicts kinked

rings and a line of contrast caused by the stacking faults in this case.

Thus, we can conclude that the dislocation reaction given by Eqn. 8 is not inconsistent with our observations. In order to substantiate this claim further it might be worthwhile to employ the computer simulation technique^(31,32) of dislocation contrast effects. Though it is clear that any further analysis will not change the general picture that we have described.

5.2 The effect of the electric field induced stress on the stability of stacking faults

The high electric field used to field evaporate and/or image an FIM specimen results in a stress distribution which has yet to be calculated exactly; although Smith and Smith⁽⁴²⁾ have calculated an average value of σ_{zz} [the stress which acts on the z-plane along the z-axis (the specimen axis)], and have found that this stress is essentially a constant value for the first $10^3 \text{\AA}$ from the specimen's surface (for a specimen with a tip radius of approximately $1000 \text{\AA}$). In the case of molybdenum a negative hydrostatic pressure (p) of $9 \cdot 10^{10} \text{ dyne cm}^{-2}$ (in Section 5.1) will result in a volume increase $(\frac{dV}{V} \cdot 10^2)^{\dagger}$ of approximately 2.6%. Vitek⁽⁴³⁾ has suggested that this large expansion of the crystal will cut off the interaction between atoms very close to second-nearest neighbors and that this would result in stable stacking faults on the {110} and {211} planes in a b.c.c. crystal. He suggests that this argument may explain the dissociated dislocations observed by Smith and Bowkett⁽⁴⁴⁾ and Smith and Gallot⁽⁴⁵⁾ in tungsten and iron respectively; although in neither of these b.c.c. metals was there any evidence found for the three fold symmetric dissociation of $\frac{1}{2}a\langle 111 \rangle$ screw dislocations

[†] Calculated from the expression⁽⁴⁶⁾ $\frac{dV}{V} = -\frac{3(1-2\nu)}{E}p$ where ν is Poisson's ratio and E is Young's modulus.

reported here. The dislocations observed by Smith and co-workers in tungsten and iron were most likely not introduced as a result of the FIM specimens yielding in-situ to the electric field induced stresses at cryogenic temperatures. These metals do not usually deform plastically in the FIM, but instead fail catastrophically after which the resulting FIM image is unusable. Hence, the dislocations they observed were most likely produced at an elevated temperature during the fabrication of the wire specimens by a drawing process.

Since there are no detailed calculations yet available to predict the effect of the field-induced stresses on a given dislocation configuration in an FIM specimen we wish to suggest that it would be very worthwhile to examine the effects of large negative hydrostatic pressures on the stability of the dislocation core structure of $\frac{1}{2}a\langle 111 \rangle$ screw dislocations that exhibit three fold symmetric dissociation employing discrete lattice calculations. It is certainly possible that the field induced stresses play an important role in stabilizing the three fold symmetric dissociation of the $\frac{1}{2}a\langle 111 \rangle$ screw dislocations that we have observed, but it is not yet clear as to how this stabilization occurs.

5.3 Separation of partial dislocations

In Section 4.3 (also see Tables 3 and 4) it was shown that there was a measureable separation between the central partial dislocation and the three partial dislocations which terminated the $\{110\}$ stacking faults. It was noted that the separation between partial dislocations was a function of the local radii and that this separation decreased as the local radii decreased. Let us now consider the question of the observed separation of partial dislocations

at a surface.

Gevers, Amelinckx and Delavignette⁽⁴⁷⁾ have considered the energetics of the problem of the intersection of a dissociated dislocation with a flat free surface. They have examined four possible situations[†] (see Figs. 1 and 2 in the Gevers et al.⁽⁴⁷⁾ paper) for the case where a stacking fault bounded by two partial dislocations intersects a flat surface which is assumed to be atomically clean. Their diagrams show that it is possible to have either an expansion or contraction of the distance between the partial dislocations bounding the stacking fault. This expansion or contraction effect varies as a function of distance from the point where the partial dislocations intersect the free surface. The four different possible topologies are determined by the sign and the magnitude of $\hat{n} \cdot \vec{b}_p$ for each of the partial dislocations bounding the stacking fault. They employed the calculus of variations to find the configuration which minimized the energy difference between the original bulk and considered configurations. Under the assumption of isotropic elasticity theory they considered the following contributions to the energy of the different configurations: (1) the line energy of the dislocations; (2) the stacking fault energy; (3) the surface energy (assumed to be a constant) of the ledge produced; (4) the interaction between the partials bounding the stacking fault; and (5) the interaction between successive stacking faults of a sequence. Their calculations neglected any effects of image forces on the partial dislocations. The final expressions they obtained

[†] It is noted that case (d) in their figures involves a second dissociated dislocation lying in the same slip plane and which precedes the first dissociated dislocation and also has a different total Burgers vector.

relate the surface energy and the stacking fault energy to a number of measureable geometric properties of the dissociated dislocations.

Our case is considerably more complicated than the situation envisioned by Gevers et al. for the following reasons: (1) the large negative hydrostatic stress acting on the tip may affect the equilibrium separation distance between the partial dislocations; (2) the energy of the ledge produced where the stacking fault intersects the surface is affected by the small radii of curvature of a sharply pointed FIM tip; and (3) the configuration shown in Fig. 8 involves the interaction of three stacking faults and four dislocations.

We have already discussed point (1) in Section 5.2 and will not pursue it any further. Let us now consider point (2). Within the context of a continuum thermodynamic model the chemical potential of an atom (μ_a) at a point on a curved surface[†] is given by the Herring equation⁽⁴⁸⁾

$$\mu_a = p\Omega_a + \left[\gamma \left(\frac{1}{r_1} + \frac{1}{r_2} \right) + \frac{\partial^2 \gamma}{\partial n_x^2} \cdot \frac{1}{r_1} + \frac{\partial^2 \gamma}{\partial n_y^2} \cdot \frac{1}{r_2} \right] \Omega_a, \quad (9)$$

where Ω_a is the atomic volume of an atom, γ is the surface tension, r_1 and r_2 are the principal radii of curvature of the original surface, and n_x and n_y are the projections of the variable unit vector $\hat{n}$ into a plane tangent to the surface at the point under consideration. The x-axis being chosen in the direction of the principal curvature $1/r_1$ and the y-axis in that of $1/r_2$. Equation 9 shows that the chemical potential of an atom in a ledge in a surface

[†] We have taken the chemical potential of an atom on a flat surface to be zero.

with small values of r_1 and r_2 will be greater than that of an atom in a ledge on a surface with larger values of r_1 and r_2 . Thus the energy of a ledge* of a fixed length increases as r_1 and r_2 decreases. This qualitative argument implies that as r_1 and r_2 are decreased the same ledge would decrease its length to help minimize the overall free energy of the configuration; i.e., the separation between the partial dislocations would be smaller in the specimen with the smaller values of $r_{(222)-(244)}$. This conclusion is not meant to imply that in an unstressed bulk specimen the equilibrium spacing between the partial dislocations would be greater than the values reported in Table 3, rather it is intended to explain our specific FIM† observations.

In view of the complexity of the present situation it does not seem reasonable to try to extract a value of the stacking fault energy from our measurements of the separation between the partial dislocations. Although Smith et al. (49) have used a simple expression, derived by Gilman (50) for the contraction of a dissociated screw dislocation which emerges normal to a free surface, to calculate the stacking fault energy for a dissociated dislocation observed in an Iridium FIM specimen; we do not feel that this calculation is warranted at the present time because of the problems discussed in this section and in Section 5.2.

* The actual energy of the ledge should be obtained from an atomistic model that involves the details of the atom positions in a specific ledge on a highly curved surface. In this paper we are using a continuum thermodynamic model which is embodied in Eqn. 9 as a first order treatment of this problem.

† It is noted that the specimen with the smaller values of $r_{(222)-(244)}$ (see Table 4) was also the more impure one; i.e., the one with $R = 33$.

5.4 Possible Suzuki segregation of impurity atoms to stacking faults

It is felt that Suzuki impurity segregation effects⁽⁵¹⁾ are not the cause of the measured separation distances between the partial dislocations; i.e., the measured distances are not caused by the preferential adsorption or desorption of solute atoms on or from the stacking fault ribbons, which results in a decrease of the stacking fault energy and a subsequent increase in $r_{(222)-(244)}$. There are two points which we wish to make to substantiate the above statement.

The first point is that the FIM specimens were deformed by the electric field induced stresses at T_T 's of approximately less than 50 K. This implies that the solute atoms which were adsorbed or desorbed on or from the stacking faults would have had to be mobile at a T_T of less than 50 K. This requirement does not seem highly probable.

The second point concerns an estimate of the maximum value of the degree of coverage of the stacking fault ribbons by adsorbed impurity atoms. For the R equal $2.2 \cdot 10^3$ specimen (see Table 3) the degree of coverage would be approximately $3 \cdot 10^{-3}$ of a monolayer, if all the impurity atoms* in the FIM tip ($200\text{\AA}$ average radius) were adsorbed on the three stacking fault ribbons associated with the dissociated $\frac{1}{2}a\langle 111 \rangle$ screw dislocation. A similar calculation for the R equal 33 specimen (see Table 4) yielded a maximum value of the degree of coverage of 0.07 of a monolayer. If Suzuki segregation effects were important in our specimens then we would expect

* The calculations were made employing the impurity atom concentrations determined from the measured values of the 4.2 K resistivity, since these values represent the impurity atom concentrations in solid solution.

the separation distance between the partial dislocations to be larger in the R equal 33 specimen as opposed to the R equal $2.2 \cdot 10^3$ specimen. In fact just the opposite effect was observed (compare the values in Tables 3 and 4).

6. SUMMARY AND CONCLUSIONS

1. The first observation of a three fold symmetric dissociated screw dislocation structure in any b.c.c. metal has been detected in molybdenum specimens which were deformed by the electric field induced stresses, that must act on the tip region of all FIM specimens, at a specimen temperature of less than 50 K.
2. The structure was always observed in the $\{222\}$ planes and consisted of a partial $\langle 111 \rangle$ type screw dislocation emerging normal to a $\{222\}$ type plane with stacking faults lying on the three $\{110\}$ planes which intersected one another along a $\langle 111 \rangle$ type direction (see Fig. 8). The central partial dislocation was of the form $ka\langle 111 \rangle$ where k is a constant. The three $\{110\}$ stacking faults exhibited three fold symmetry about the central $ka\langle 111 \rangle$ type partial screw dislocation.
3. The separation distance between the central partial dislocation ($ka\langle 111 \rangle$) and each partial dislocation which bounded the stacking faults on the $\{110\}$ planes was a function of the local radii of curvature of the tip (see Tables 3 and 4). A first order model was presented to explain this effect.
4. The observed three fold symmetric dislocation structure was present in both low purity (approximately $1.2 \cdot 10^{-3}$ at. fr. impurity

content) and high purity (approximately $1.7 \cdot 10^{-5}$ at. fr. impurity content) molybdenum specimens. Simple arguments were presented to show that the observed dislocation dissociations could not have been caused by Suzuki segregation effects.

5. The basic three fold symmetric dissociated dislocation structure persisted through continuous and extensive field evaporation of the specimens. In addition, elements of the contrast effects reported in this paper can be seen in the $\{222\}$ planes and environs of all of the single frame micrographs of molybdenum published in the literature.

6. It is suggested that the effects of large negative hydrostatic pressures (greater than and equal to $9 \cdot 10^{10}$ dyne cm^{-2}) on the stability of the dislocation core structures of $\frac{1}{2}a\langle 111 \rangle$ screw dislocations, that exhibit three fold symmetric dissociation, be examined employing discrete lattice calculations.

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Table 1. Spark source mass spectrometric analysis of a molybdenum specimen with R equal to 33.

Impurity	Co	Rb	Nb	Mg	As	Ca	Mn	P	S	W	Al	Si	K	Cu	Fe	N ^(b)	O ^(c)
Concen- tra- tion ^(a) x(10 ⁻⁶ at.fr.)	0.1-1	0.1-1	0.1-1	1- 10	1- 10	1- 10	1- 10	1- 10	1- 10	4- 40	10- 100	10- 100	10- 100	10- 100	100- 1,000	<1,000	3975

- (a) Standards were not available for molybdenum, so the concentrations reported were estimated assuming that all species were ionized to the same degree by the ionizing spark. Generally, this procedure produces results that are accurate to within a factor of two to three.
- (b) These concentrations were reported as upper limits, due to a possible interference in their determination.
- (c) A value of 3975 ppm for oxygen was obtained via a wet chemical test after the spark source spectrometric analysis was performed for the other elements.

Table 2. Spark source mass spectrometric analysis of a molybdenum specimen with R equal to 2200.

Impurity ^(c)	Mn ^(b)	P ^(b)	Fe	Al	Mg	Ca	W	Si	N ^(b)	O ^(d)	C ^(b)
Concentration ^(a) (x10 ⁻⁶ at.fr.)	<1	<1	1-10	1-10	1-10	1-10	4-40	10-100	<100	312	<1000

- (a) Standards were not available for molybdenum, so the concentrations reported were estimated assuming that all the species were ionized to the same degree by the ionizing spark. Generally, this procedure produces results which are accurate to within a factor of two to three.
- (b) These concentrations were reported as upper limits due to a possible interference in their determination.
- (c) The F, Na, Ti and Hg concentrations were unavailable due to interference.
- (d) A value of 312 ppm for oxygen was obtained via a wet chemical test after the spark source spectrometric analysis was made for the other elements.

Table 3. Local tip radii and separation between partial dislocations for specimen shown in Fig. 5.†

Plane in which second partial dislocation is observed. $\{hkl\}$	Local tip radii between (222) and (244) planes $r_{(222)-(244)}$ $(\text{\AA})^\dagger$	Separation between partial dis- locations. ρ_i $(\text{\AA})^*$
(244)	193±24	56±7
(442)	217±24	68±7
(424)	217±24	63±7

† This specimen had an R value of $2.2 \cdot 10^3$.

† Calculated from Eqn. 5.

* Calculated employing Eqn. 6.

Table 4. Local tip radii and separation between partial dislocations for specimen shown in Fig. 6.†

Plane in which second partial dislocation is observed. (hkl)	Local tip radii between (222) and {244} planes $r_{(222)-(244)}$ (Å) [†]	Separation between partial dislocations. ρ_i (Å) [*]
(244)	145±24	43±7
(442)	121±24	36±7
(424)	121±24	36±7

† This specimen had an R value of 33.

+ Calculated from Eqn. 5.

* Calculated employing Eqn. 6.

FIGURE CAPTIONS

- Figure 1. A schematic illustration of the contrast produced by a stacking fault which is characterized by a constant shear vector $\vec{R}$ and a unit normal to the plane of the fault $\hat{s}$. Figure 1a is a cross-sectional view of a single pole on the surface of an FIM tip which is characterized by a unit normal $\hat{n}$. Figure 1b is the same pole after the stacking fault has been introduced. In this case q is non-integral and lies between zero and unity. Figure 1c is a top view of Fig. 1b after the cross-hatched area in Fig. 1b has been removed by field evaporation. The lines of contrast are parallel to the vector $\hat{n} \times \hat{s}$ and these lines shift to the left as field evaporation of the crystal proceeds. If the vector $\vec{R}$ was parallel to $\hat{n}$ then the line of contrast would not shift to the left as field evaporation proceeded.
- Figure 2. The top view of a pole on field ion microscope tip which contains a single partial dislocation that bounds a stacking fault. The case illustrated here is for $0 < |q| < 1$. This contrast effect is termed a single stepped spiral.
- Figure 3. A partial standard 222 stereographic projection. The planes that are labeled are the ones that are relevant to the contrast effects presented in Figs. 4, 5 and 6. The traces of the three $\{110\}$ planes which contain the observed stacking faults are also indicated in this projection.

Figure 4. The portion of the surface of a molybdenum FIM specimen ($R = 2.2 \cdot 10^3$) which corresponds to the partial 222 stereographic projection shown in Fig. 3. This micrograph was recorded at a T_T of 12.7 K.

Figure 5. A pulse field evaporation sequence of the (244) and (233) planes. The line of contrast that runs through these planes is the trace of the $(01\bar{1})$ plane. These 10 frames were selected from a total of 181 frames of ciné film which were recorded between frame numbers 1 and 181. The vees point to the presence of steps in the (233) planes. A schematic is presented below each numbered frame. This specimen had an R value of $2.2 \cdot 10^3$. See Section 4.2 for a detailed discussion of this figure.

Figure 6. A pulsed field evaporation sequence showing 10 frames of ciné film out of a sequence of 224 frames of film. This figure exhibits the region of the FIM surface which is indicated in Figs. 3 and 4. The three lines of contrast caused by the $(01\bar{1})$, $(\bar{1}10)$ and $(\bar{1}01)$ planes in the (233), (332) and (323) planes respectively can be observed by examining frame numbers 14, 47, 84 and 128. The vees in frame numbers 1 and 84 point to the steps in the outermost ring of atoms in the (222) plane. For a schematic illustration of this effect see Fig. 7. This specimen had an R value of 33.

Figure 7. A schematic illustration of the three steps detected in the $\{222\}$ plane shown in Fig. 6. The dashed lines indicate the traces of the three $\{110\}$ planes which contain the observed stacking faults. A partial screw dislocation of the form $ka[111]$, where k is a constant, lies at the intersection of these three planes.

Figure 8. A schematic illustration of the dislocation configuration observed in the field-ion micrographs presented in Figs. 4, 5 and 6. The partial dislocations indicated in this diagram are described by a dislocation reaction of the form given by Eqn. 7.

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.

Figure 7.

Figure 8.