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Final Atomic Energy Commission Report
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A STUDY OF DISLOCATION MOBILITY AND DENSITY
IN METALLIC CRYSTALS

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I. INTRODUCTION

This report summarizes the research accomplishments under the Atomic Energy Commission contracts, CALT-473 and CALT-767-P3 for the ten-year period, November 1, 1963 to October 31, 1973. The research was stimulated by technological advances which required improvements in our ability to predict the deformation behavior of materials.

In the mid 1930's, theoreticians first recognized that crystal defects could play a central role in plastic deformation, and since that time a number of experiments have conclusively demonstrated the one-to-one correspondence between the motion of line defects (dislocations) and plastic deformation. Before the existence and significance of dislocations was recognized, theoreticians faced a puzzling problem: the predicted strength of crystals was several orders of magnitude larger than the strength actually observed. With the realization that crystal deformation is caused by the motion of dislocations, the theoretical problem reversed. The new problem became one of understanding the origin of the resistance to dislocation motion in order to explain the observed strength of crystals.

The Atomic Energy Commission Sponsored Research on dislocation mobility and density in metallic crystals at the California Institute of Technology has focused on an understanding of the dynamics of dislocations. Important interactions between a moving dislocation and lattice phonons, conduction electrons, other dislocations, and point defects such as those introduced by neutron irradiation have been studied.

The experimental phase of this research involved the introduction of isolated dislocations into a crystal, the observation of these dislocations by chemical or electrolytic etching, X-ray topography, and transmission electron microscopy (TEM); the application of appropriate stresses of controlled amplitude and duration, and finally determination of the stress-induced motion of the dislocations by observation of their new locations. The nature of the resistance to dislocation motion is deduced from these experiments.

II. EXPERIMENTAL DEVELOPMENTS

We have developed a unique capability for experimental studies of the behavior and structure of isolated crystal defects. These capabilities include (i) special facilities for the growth of crystals of high structural perfection, and for acid and electrolytic machining of suitable test specimens (ii) means of selectively introducing isolated dislocations into the test specimens (iii) X-ray and TEM facilities for observation of the defects, and (iv) special dynamic loading systems for producing single, short duration stress pulses (torsional and compressional stress states). These capabilities have been used for:

- 1) making direct measurement of the drag force on moving dislocations in close-packed crystals for velocities up to about one-tenth the shear wave velocity and at temperatures between 4.2°K and 400°K . These measurements are unique in that they are made under conditions where dislocation-dislocation interactions are negligible so that dislocation-phonon and dislocation-electron interactions may be determined. Dislocation interactions with

phonons and electrons influence the stress-strain response of crystals at the very high deformation rates which occur under conditions of shock loading.

- 2) determination of the stress-velocity relationships for dislocations in body-centered-cubic crystals. The first direct measurements of screw dislocation velocities in BCC metals (silicon-iron) and of edge dislocation velocities in high-purity iron crystals were made with these capabilities. The very significant differences in dislocation mobility in body-centered-cubic crystals and in face-centered-cubic and hexagonal crystals (on close-packed planes) were first demonstrated by these measurements.
- 3) measurement of the strength of dislocation-dislocation interactions. The first direct measurement of the critical stress required to drive a dislocation on one slip plane through a forest of dislocations which intersect the slip plane has been made, and the stress-velocity relationship at stresses above the critical stress has been determined. Analysis of these measurements has given information about the strength of individual dislocation-dislocation interactions as well as their combined effect on dislocation mobility.
- 4) measurement of the total line energy of a screw-oriented dislocation parallel to and near a free surface of the crystal. The energy contained in the dislocation core (where continuum linear elasticity theory breaks down) is deduced from this measurement.

- 5) measurement of the strength of dislocation-point defect interactions. Direct measurements of the mobility of dislocations through a field of defects gives the strength of the interactions and the resulting applied stress-dislocation velocity relationship which is fundamental to an understanding of irradiation hardening.
- 6) analyses of defect species and position. We have developed theoretical and numerical techniques to not only assist in the identification of defects by diffraction contrast techniques (e.g. X-ray topography and transmission electron microscopy), but also locate the defect accurately with respect to its associated diffraction contrast image. We have used these techniques to carry out stereography and interaction studies.
- 7) analyses of defect ultrastructure. Weak beam and Fourier synthesized electron micrographs of lattice defects potentially contain information about the atomic structure of the defect. Quite often, however, this information is obscured due to noise sources such as instabilities in the electron optics. We have developed systems, based on computer image processing, to reduce image noise. The associated increase in image resolution, as a result of computer processing, will potentially allow us to perform detailed studies on such important problems as the atomic structure of a dislocation core.

INFORMATION PRODUCTS

A total of sixty-six scientific and technical reports have been issued to the Atomic Energy Commission, and over forty publications in books and technical journals have appeared in the open literature giving acknowledgement to the Atomic Energy Commission for research support. A listing of these reports is presented in Appendix I.

Our most significant contributions to the technical literature in five different areas are summarized below:

- 1) Dynamic testing systems for the application of microsecond duration stress pulses. CALT-473-1, and CALT-767-P3-14.
- 2) Dislocation observation by X-ray topography. CALT-473-22, CALT-767-P3-3, 19, 25 and 34.
- 3) Mobility of isolated dislocations on close-packed planes of:
Cu - CALT-473-13, 14, CALT-767-P3-5, 8, 16 and 21.
Al - CALT-473-29 and 31.
Zn - CALT-473-15, CALT-767-P3-4, 16 and 17.
- 4) Mobility of slip bands in:
Zn (Basal) - CALT-473-4, 5, 16 and 17.
Zn (Second-order pyramidal) - CALT-473-6, 18 and 26.
Silicon-iron - CALT-473-20 and 23.
Fe - CALT-767-P3-7 and 15.
- 5) Diffraction calculations - CALT-767-P3-22 and 26.

III. RESEARCH PERSONNEL

Co-principal investigators for the period November 1, 1962 to October 31, 1970 were Professors T. Vreeland, Jr. and D.S. Wood. For the period November 1, 1970 to October 31, 1973, Professors T. Vreeland, Jr. and R. E. Villagrana were co-principal investigators. The following post-doctoral personnel were associated with the research effort:

Dr. Kenneth H. Adams (Now Associate Professor)
Tulane University).

Dr. David P. Pope (Now Associate Professor,
University of Pennsylvania).

Dr. Arthur P.L. Turner (Now Assistant Professor,
Massachusetts Institute of Technology).

Dr. Kenneth M. Jassby (Now Senior Lecturer,
University of Tel-Aviv).

Dr. Norio Nagata (Now Staff Scientist, National
Research Institute for Metals, Tokyo, Japan).

Dr. Silvanus S. Lau (Now Bechtel Instructor,
California Institute of Technology).

Eight Ph. D. degrees were granted to students whose research was supported under CALT-473 and CALT-767-P3. An average of five undergraduate students per year gained research experience through part-time work under the Atomic Energy Commission sponsorship.

IV. ANNUAL REPORT FOR THE FINAL YEAR

IV.1 Dislocation Interactions with Irradiation Induced Defects

The experiments were designed to measure:

1) The critical stress required to drive a fresh basal dislocation through the neutron irradiated crystals,

2) The velocity vs. stress relationship for the fresh basal dislocations at stresses above the critical stress, and

3) The type and density of irradiation induced defects.

Six single crystal cylinders of zinc were prepared ([0001] cylindrical axis, 0.5 inch diameter, approximately 0.5 inch long) and shipped to Dr. T.H. Blewitt at the Argonne National Laboratory for irradiation. The crystals were given the following treatment:

Average Neutron Energy = 0.8 MEV

Temperature during irradiation 4.2°K to 5°K

Crystal*	10^{16} Neutrons/cm ²	Date of Irradiation
1	84	9/09/71
3	19	10/28/71
4	19	10/28/71
5	6.8	11/11/71
6	6.8	11/11/71

*Crystal #2 was damaged in shipping.

The crystals exhibited a maximum activity after irradiation of 165 mr/hr (at 2 inches), which was attributed to the isotope Zn⁶⁵ (half life = 243 days). The experiments to be performed on the zinc crystals required that they be handled for sufficiently long periods that radiation exposure was a problem. The crystals were aged at room temperature for approximately 600 days before the experiments began, and the activity had then dropped to about 15 mr/hr. This activity was sufficiently low to permit specimen

handling for the time required without exceeding the radiation dose set by the California Institute of Technology Radiation Safety Committee.

The crystals were attached to one-half inch diameter cylinders of polycrystalline titanium of various lengths using a thin layer of epoxy. The titanium cylinders, coaxial with the $[0001]$ axis crystals, were useful for handling purposes, and their length together with the length of the crystal determined the loading duration at the end (0001) observation surface of the crystal when it was coupled to the torsion pulse loading system (CALT-473-1).

The (0001) observation surfaces of the zinc crystals were badly oxidized after the irradiation and room temperature aging, so that chemical lapping was required to remove the oxide and give a clean, flat (0001) surface suitable for dislocation observation by X-ray topography. Unfortunately, this lapping removed the surface which had been annealed (prior to the irradiation). The basal dislocation density within about 50μ of an annealed surface is essentially zero, while the density several hundred microns below an annealed surface is about 10^4 cm/cm^3 which is the bulk, as-grown dislocation density. The lapping operation thus gave us a background dislocation density of about 10^4 cm/cm^3 , which complicated interpretation of the test results.

Fresh basal edge dislocation were introduced by scratching the crystal surface (200 mg. to 250 mg. loads) with a filament of quartz of about 20μ diameter. Three diametral scratches in $[\bar{1}010]$, $[0\bar{1}10]$, and $[1\bar{1}00]$ directions were made. Prior to scratching, a grid of 38μ diameter gold dots, about 0.1μ thick, was vacuum deposited onto

the test surface of some specimens to serve as a reference. Two X-ray topographs (using $(10\bar{1}3)$ and $(1\bar{1}03)$ reflections) were taken prior to scratching to locate the background dislocations.

Within thirty minutes after scratching, the test surface of the crystal was coupled to the torsion testing system using a glycerine-ethanol mixture (1 : 46) and the specimen section of the system was cooled to 103°K where the glycerine-ethanol mixture is sufficiently viscous to pass the torsional stress pulse. A single torsional stress pulse was then applied to the test surface of the crystal. The amplitude τ_{max} and duration of the pulse were deduced from the output of a strain gage bridge attached to the one-half inch diameter titanium rod of the torsion pulse system and located one inch from the test surface.

After the stress pulse was applied and the system was warmed to room temperature, two $\{10\bar{1}3\}$ X-ray reflection topographs were taken of the test surface. The topographs typically showed dislocation motion to have occurred away from the scratch near the periphery of the specimen where the applied stress was maximum, with no motion near the center where the stress was lower.

IV 1.1 Critical Stress Measurements

The minimum radius at which motion occurred (r_c) was measured and the critical stress calculated from the relation

$$\tau_c = \frac{r_c}{r_o} \tau_{\text{max}} \quad (1)$$

where r_o = outer radius of the specimen. Equation 1 is valid provided the plastic strain in the specimen is small compared to the elastic strain.

This condition may be expressed as:

$$\gamma_p = \rho \ell b \ll \gamma_e = \frac{\tau_o}{c_{44}} \quad (2)$$

where ρ = mobile dislocation density (about 10^4 cm/cm^3 initially)

ℓ = maximum dislocation displacement (about .05 cm)

b = Burgers vector ($2.66 \times 10^{-8} \text{ cm}$)

c_{44} = shear modulus ($3.88 \times 10^{11} \text{ dyn/cm}^2$)

γ_e = elastic strain

Thus, the plastic strain was about 1.4×10^{-5} which is smaller than the elastic strain at r_o (minimum $\gamma_e = 6 \times 10^{-5}$), and equation 1 is a good approximation for the initial tests. The dislocation density increases somewhat in each test, and after nine tests (the maximum number for any one specimen) the plastic strain could be comparable to the elastic strain. It was observed that significant density increases occurred only in the outer twenty percent of the radius. Assuming an elastic-perfectly plastic material behavior, the elastic stress in the region of the critical radius is increased by eight percent relative to the purely elastic case. An elastic-perfectly plastic assumption for basal slip in zinc at small plastic strain ($\gamma_p \approx \gamma_e$) is very conservative, so that equation 1 is a good approximation for all of the tests of this investigation.

Values of τ_c calculated from measurements of r_c in eighteen tests on three different crystals are given in Table I. Crystal 4 has not yet been tested, and crystal 1 was damaged in handling. We are attempting to make critical stress measurements on crystal 1. Two tests on crystal 1 have failed due to poor dislocation production in the scratching operation and to high initial dislocation density.

Critical radius measurements could not be obtained from some of the tests on crystals 3, 5 and 6 because displacements of scratch-induced dislocations were obscured by grown-in dislocations.

Crystal 3, subjected to the higher neutron dose, has a higher average critical stress than crystals 5 and 6. The relatively large scatter in critical stress values for each crystal is attributed to a nonuniform defect distribution as discussed in section IV 1.3 below.

IV 1.2 Velocity vs. Stress Measurements

The presence of grown-in dislocations contributed considerable uncertainty to the determination of displacements of the scratch-induced dislocations. In the high stress region near the periphery of the crystal, displacements and multiplication of grown-in dislocations took place, making it difficult to recognize the "front" of fresh dislocations which moved away from the scratch. Such a "front" could be recognized on several specimens, and an example is shown in Fig. 1. A plot of dislocation displacement vs. radius is shown in Fig. 2, together with the calculated displacements for a crystal which had not been irradiated (only viscous dislocation drag acting due to phonon interactions, CALT-767-P3-12). The measured displacements approach the calculated displacements at a radius about twice the critical radius. Thus, dislocation velocities become viscous drag limited (rather than defect limited) at stresses above about twice the critical stress. This same behavior was found for basal dislocations interacting with a forest of second-order pyramidal dislocations in zinc (CALT-767-P3-18).

IV 1.3 Defect Observations

The irradiated zinc specimens were studied by transmission electron microscopy and diffraction; the objective being to determine the void density. Void identification was made by a combination of contrast experiments and stereoscopic analysis.

The results of our microscopic investigations indicated that the voids existed in random clusters throughout the volume of the zinc crystals. We were unable to correlate this cluster density to neutron dosage because of the non-uniform distribution.

IV 1.4 Summary of Results

The critical stress measurements show an increase in critical stress with neutron irradiation. The scatter in measured values is attributed to a non-uniform distribution of irradiation-induced defects. Voids observed in the T.E.M. specimens were most probably the defects that strengthened the crystals. Their non-uniform distribution may be related to the non-uniform distribution of the grown-in dislocations which acted as sinks for the irradiation-induced interstitials and vacancies. The critical stress could be fitted to the relation $\tau_c \sim \sqrt[3]{N}$ where N is the neutron dose.

V. SLIP BAND GROWTH ON THE SECOND-ORDER PYRAMIDAL SYSTEM OF ZINC

Measurements of the growth of slip bands produced by compression stress pulses from approximately 10 μ sec to 100 μ sec duration have been made at 77°K. The velocity of the dislocations at the head of the slip band has been deduced from these measurements

for pure edge, pure screw, and mixed dislocations. The mobility of mixed dislocations exceeded that of the screw and edge dislocations, and this observation cannot be accounted for by the usual presumption of phonon and electron damping. Since these dislocations lie on non-close-packed planes, their motion requires extensive elongation of atomic bonds normal to the slip plane with an attendant radiation of elastic energy. The stress state acting on the mixed dislocations whose motion was observed had a lower resolved shear stress on the slip plane than the stresses acting on the crystallographically equivalent slip plane on which edge and screw dislocation displacements were observed. However, the mixed dislocations were displaced further by the stress pulse.

This effect has not been treated analytically, but such a radiation damping may give rise to the relatively large residual damping of dislocation motion on close-packed planes of copper at 4.2°K , where it has been shown that the damping is considerably larger than that predicted by the electron and phonon damping mechanisms (CALT-767-P3-21).

VI. DISLOCATION MOBILITY AT LOW TEMPERATURE IN COPPER AND LEAD

Measurements of dislocation mobility in copper at 4.2°K have been completed, and reprints of CALT-767-P3-21 are submitted with this report.

Measurements of dislocation mobility in lead crystals at 4.2°K have been attempted, but difficulty has been experienced in introducing

short isolated bands, and in observing the growth of the bands.

Berg-Barrett X-ray reflections have been found which reveal the bands, and further testing of superconducting and normal lead will be done in the future.

VII. DISLOCATION MOBILITY STUDY IN B. C. C. CRYSTALS

Dislocation mobility in molybdenum single crystals reported in the literature is characterized by a strong stress and temperature dependence.^{1,2} These results were obtained by a stress pulse-displacement technique and the motions of dislocations were revealed by etch pitting. Due to the limitations of this technique it had to be assumed that dislocations introduced into the crystal either by indenting or scratching the crystal surface were edge dislocations and that slip took place on the plane of maximum resolved shear stress. Since the X-ray topographic technique reveals considerably more detail of the slip band structure, it was the objective of the present work to investigate (1) the characteristics of slip bands introduced by various techniques, (2) the mobility of both edge and screw dislocations. It is of particular interest to measure the mobility of screw dislocations, because the deformation of molybdenum as well as other refractory b.c.c. crystals is seemingly controlled by the motion of screw dislocations.

It was found in the present work that slip bands introduced by indenting the (112) surface with a sharp tool are composed of dislocations with all four possible Burger's vector, i. e. $\frac{1}{2}[111]$, $\frac{1}{2}[1\bar{1}1]$, $\frac{1}{2}[11\bar{1}]$, and $\frac{1}{2}[\bar{1}11]$. The center portion of the slip bands consists of dislocations primarily of the $\frac{1}{2}[11\bar{1}]$ Burger's vector while the dislocations near

the two edges of the slip bands are mostly of the other three types of Burger's vectors (CALT-767-P3-19).

Fine slip bands are formed by scratching the (112) surface with a needle in a direction normal to the $[11\bar{1}]$ direction (direction of the Burger's vector lying in the (112) plane) with the needle axis almost parallel to the surface. Extinction experiments show that these slip bands are made up of dislocations with the $\frac{1}{2}[11\bar{1}]$ Burger's vector. If these bands were made up of dislocation loops lying on (112) planes parallel to the surface, then the length to width ratio of these bands implies that the mobility of edge dislocations is at least ten times greater than that of the screw dislocations at room temperature.

Scratching the (112) surface of a molybdenum crystal with a needle almost parallel to the surface and in the $[11\bar{1}]$ direction produces individual dislocations and long slip bands that are very near the surface. Extinction experiments show that these are screw dislocations with a $\frac{1}{2}[11\bar{1}]$ Burger's vector, and the slip bands consist of dislocations of the same Burger's vector.

It was thought that screw dislocation mobility could be measured by observing the displacement of the individual screw dislocations produced by the above mentioned technique. However, after stressing the crystal with a pure torque³ for periods of thirty minutes to two hours these screw dislocations disappeared presumably due to the cross-slipping of dislocations to the specimen surface.

The edge dislocation mobility has been investigated by applying a torque to the (112) surface of the specimens. Slip bands with a $\frac{1}{2}[11\bar{1}]$ Burger's vector were introduced by an indentation technique.

Measurements were made at room temperature. Resolved shear stresses covered a range of 80 - 400 M dyn/cm². Dislocation velocity (v) was found to follow a

$$v = v_0 \left(\frac{\tau}{\tau_0} \right)^m$$

relationship, where $v_0 = 1$ cm/sec and τ is the resolved shear stress on the (112) slip plane. The values of m and τ_0 were found to be 2.6 and 5.4×10^3 M dyn/cm² respectively, as compared to $m = 6.4$ and $\tau_0 = 539$ M dyn/cm² (5.5 Kg/mm⁻²) reported by Prekel et. al.

Screw dislocation mobility can be measured by the growth of the width of the slip bands. The presence of dislocations of three other Burger's vectors near the two edges of the as-introduced slip bands might interfere with accurate velocity measurements. However, when the slip bands have grown much beyond their original lengths by the motion of dislocations, it is expected that the extended slip bands are free of dislocations of other Burger's vectors and their growth in width will reflect the screw dislocation velocity. Experiments to determine the screw mobility by measurements of the growth in width of the bands is now in progress.

VIII. BERG-BARRETT STEREO TOPOGRAPHY

Developments have been made in our ability to study the depth of dislocations and other structural features by use of X-ray stereo topography.

It has been found that a grid pattern of gold, vacuum evaporated onto the crystal surface (about 0.1 μ thick) provides an excellent surface

reference in the stereo pair of topographs. Depth measurements have been made of the dislocations induced by scratching, and we have determined that the dislocations revealed in the topographs are considerably deeper than heretofore believed (about 20μ instead of 2μ). Further experiments are underway to determine the characteristics of the diffraction from dislocations at different depths in crystals of zinc and copper.

Figure 1

Berg-Barrett topograph of a scratched (0001) surface of specimen 5 after test 3, (10 $\bar{1}$ 3) reflection, CoK α radiation, 31.5X. A dense distribution of dislocations which were displaced by the torsion stress pulse may be seen above the horizontal scratch. Background dislocations complicate determination of the maximum displacements of the scratch-induced dislocations.

Figure 2

Displacement vs. Radius for the scratch-produced dislocations of specimen 5 after test 3. The dashed line shows the displacements that would have been produced by the stress pulse if the dislocation velocity was limited only by phonon drag.

TABLE 1

Critical Stress Measurements

<u>Crystal</u>	<u>Test Number</u>	<u>Critical Stress</u> <u>10^6 dyn/cm^2</u>	<u>Mean</u> <u>10^6 dyn/cm^2</u>	<u>STD Deviation</u> <u>10^6 dyn/cm^2</u>
3	2	12.1	11.34	1.09
	3	13.3		
	5	11.4		
	6	10.65		
	8	10.1		
	9	10.5		
5	1	11.2	9.9	2.36
	2	5.0		
	3	13.3		
	4	9.9		
	5	10.8		
	6	10.0		
	9	9.0		
6	1	8.4	8.4	0.84
	2	7.4		
	5	9.9		
	6	8.3		
	7	7.9		

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APPENDIX I

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- CALT-473-2 "Etching of High-Purity Aluminum", by W.F. Greenman. Submitted to AEC: August 1964.
- CALT-473-3 "A Study of Dislocation Mobility and Density in Metallic Crystals", by T. Vreeland, Jr. and D.S. Wood. Submitted to AEC as Annual Summary Report #3: July 1965.
- CALT-473-4 "Basal Dislocation Mobility in High-Purity Zinc Single Crystals", by K.H. Adams, T. Vreeland, Jr. and D.S. Wood. Submitted to AEC: June 1965.
- CALT-473-5 "Impurity Effects on Basal Slip in Zinc", by K.H. Adams, T. Vreeland, Jr. and D.S. Wood. Submitted to AEC: July 1965.
- CALT-473-6 "Second-Order Pyramidal Slip in Zinc", by K.H. Adams, R.C. Blish, II, T. Vreeland, Jr. and D.S. Wood. Submitted to AEC: January 1966.
- CALT-473-7 "A Comparison System for Microscope Images", by D.P. Pope, T. Vreeland, Jr. and D.S. Wood. Submitted to AEC: October 1965. Published in: Review of Scientific Instruments 37, 3, March 1966, page 377.
- CALT-473-8 "Damage Due to Electric Spark Discharge Machining of Zinc", by A.P.L. Turner, K.H. Adams, and T. Vreeland, Jr. Submitted to AEC: November 1965. Published in: Materials Science and Engineering 1, 1, May 1966, page 70.
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- CALT-473-10 "A Study of Dislocation Mobility and Density in Metallic Crystals", by T. Vreeland, Jr. and D.S. Wood. Submitted to AEC as Annual Summary Report #4: July 1966.
- CALT-473-11 "Dislocation Mobility in Pure Copper Single Crystals", by W.F. Greenman, T. Vreeland, Jr. and D.S. Wood. Submitted to AEC: December 1966.

- CALT-473-12 "Dislocation Velocity Measurements", by T. Vreeland, Jr. Submitted to AEC: January 1967. Published in: Techniques of Metals Research, R. Bunshah, editor, Wiley & Sons.
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- CALT-473-21 "A Study of Dislocation Mobility and Density in Metallic Crystals", by T. Vreeland, Jr. and D.S. Wood. Submitted to AEC as Annual Summary Report #5: July 1967.

- CALT-473-22 "Experimental Techniques for Observing Dislocations by the Berg-Barrett Method", by A. P. L. Turner, T. Vreeland, Jr. and D. P. Pope. Submitted to AEC: August 1967. Published in: *Acta Crystallographica* A24, (1968), page 452.
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