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DISLOCATION MECHANISMS IN THE DEFORMATION OF HIGH PURITY TANTALUM AT LOW TEMPERATURES

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### **Author**

Ranji, Sampath.

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### UNIVERSITY OF CALIFORNIA

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# DISLOCATION MECHANISMS IN THE DEFORMATION OF HIGH PURITY TANTALUM AT LOW TEMPERATURES

Sampath Ranji

(M.S. Thesis)

January, 1966

# DISLOCATION MECHANISMS IN THE DEFORMATION OF HIGH PURITY TANTALUM AT LOW TEMPERATURES

#### Sampath Ranji

Inorganic Materials Research Division, Lawrence Radiation Laboratory, and Department of Mineral Technology, College of Engineering, University of California, Berkeley, California

January, 1966

#### ABSTRACT

The object of the investigation was to determine the rate controlling mechanism for slip in body centered cubic tantalum, at low temperatures [T < 0.2 Tm].

High purity single crystals of tantalum, oriented for single slip were grown by electron beam zone refining. These crystals were tested in an Instron tensile machine to determine the temperature and strain rate dependence of the flow stress. The temperature range studied was 4.2° to 550°K, at two strain rates differing by about a factor of 100.

At low temperatures the effect of both the temperature and strain rate could be rationalized satisfactorily in terms of the Peierls' mechanism, when the deformation is controlled by the nucleation of pairs of kinks [the Dorn-Rajnak model].

#### INTRODUCTION

In recent years, considerable interest has been focused on the mechanical properties of refractory metals, chiefly Ta, Mo, Nb and W.

In addition to their refractory nature the common feature of these metals is that they crystallize in the body centered cubic structure.

The flow stress of b.c.c. metals at low temperatures [T < 0.2 Tm] is markedly dependent on both temperature and strain rate. This feature distinguishes them from the close packed structures (f.c.c. and h.c.p.).

It has been recognized that a basic understanding of the mechanical behavior of metals will in a large measure depend on an understanding of the generation, motion and interactions between dislocations and the factors which influence them. Several thermally activated mechanisms have been proposed to account for the strong temperature and strain rate dependence of the flow stress of b.c.c. metals. In chronological order, these are:

- (1) Breaking away from an interstitial atmosphere
- (2) Overcoming the Peierls'-Nabarro stress
- (3) Nonconservative motion of jogs
- (4) Overcoming interstitial precipitates
- (5) Cross slip

Conrad has made a critical review of the experimental evidence from the standpoints of activation volumes and activation energies. He concluded that the thermally activated overcoming of the Peierls'-Nabarro stress was the most satisfactory mechanism to account for the strong temperature and strain rate dependence of the flow stress.

A similar conclusion was reached by Christian and Masters. 2

The aim of the present investigation was to determine the rate controlling mechanism for slip in high purity tantalum at low temperatures. The mechanical properties of polycrystalline tantalum have been recently summarized by Tietz. Tantalum is unique among the b.c.c. transition metals in that it is ductile at ordinary strain rates at as low as 4.2°K. In order to obtain a fundamental knowledge of the low temperature behavior, it is essential to carry out tests on material of the highest purity possible. It is well known that interstitial impurities have a very strong influence on the flow stress of b.c.c. metals, especially at low temperatures. It is also essential to use oriented single crystals for the study, since the presence of grain boundaries can lead to ambiguity. Such a study of high purity single crystals should then provide an answer to the question of the rate controlling mechanism. The method of electron beam melting is eminently suited to prepare test specimens for such an investigation.

It is known that both the flow stress and work hardening characteristics of tantalum vary widely with orientation. Hence, it becomes necessary to measure flow stresses for one specific orientation of all the crystals. This orientation should be such as to permit a significant amount of plastic deformation to take place on a single slip system.

Various analyses of the Peierls' mechanism have been formulated by Seeger, 5 Lothe and Hirth and Dorn and Rajnak. 7 The experimental results of the deformation of the intermediate phases AgMg and Ag2Al, and Fe-2% Mn alloy 8-10 have been satisfactorily correlated with the Dorn-Rajnak

model of nucleation of kink pairs. It will be shown that the effect of both strain rate and temperature on the flow stress of pure Ta over the low temperature range can be completely rationalized in terms of the Dorn-Rajnak model.

#### EXPERIMENTAL PROCEDURE

Single crystals of Ta needed for the investigation were grown by the method of electron beam melting using the floating zone technique. An MRC electron beam zone refiner was used for this purpose. Oriented single crystals of length 8 in. and diameter 1/4 in. could be grown with ease, under a vacuum of 10<sup>-6</sup> torr, starting with tantalum rods of commercial purity.

The crystals were usually grown by 3 passes at a speed of 4 mm/min. The crystal was seeded in the final pass. The power input required to melt the 1/4 in. dia. rods was about 1.1 kw. This could be achieved by various combinations of beam voltage and beam current. However, it was found that a beam voltage of 6 kv and a beam current of 180 milliamps were the optimum settings from the standpoints of uniformity of diameter of the zone melted rod, and filament life.

In a few instances, when the purity of the starting material was not good, difficulties were experienced in the first fusion pass, owing to the large amount of gaseous impurities that were liberated. This surge had the effect of increasing the pressure in the chamber above the usual operating vacuum of 10<sup>-6</sup> torr. Also, the beam current would begin to rise due to deposition of the volatalized impurities on the tungsten filament. In extreme cases, this led to short circuiting of the filament. To overcome these problems, it was found beneficial to make two fast degassing passes just below the melting point, before actually melting the rod. During the degassing runs, there was less violent liberation of impurities, and the vacuum system could pump out

the gases fast enough.

The crystals were cut with a jeweler's saw without causing undue damage. In order to check the single crystallinity of the crystals, they were etched in a solution containing equal parts of conc. HF and conc. HNO<sub>3</sub>. The absence of grain boundaries was a good indication of single crystallinity. Back reflection laue photographs were then taken along the longitudinal axis of the as grown crystals. This was to verify if the crystals had attained the orientation of the seed. In all cases, the crystal seeding was better than \$20\$. The orientation of the crystals, plotted in the standard stereographic triangle, is shown in Fig. 1.

The tensile axis lies on the great circle joining the (101) and (111) poles. It is a safe assumption that the operative slip system is (101) [111]. Further, the deformed specimens when viewed through the microscope showed clear markings of single slip.

Tensile specimens were prepared from the rods by machining. The specimens were threaded at both ends to match the threads in the grips for Instron testing.

In order to remove the surface damage caused by machining, as well as to obtain a metallographic surface for subsequent studies of deformation traces, the specimens were all electropolished. The composition of the polishing solution was 80% conc. H<sub>2</sub>SO<sub>4</sub> and 20% conc. HF, using a stainless steel cathode. The anode current density was 0.75 amps/in<sup>2</sup>. The threads at the ends of the specimen were protected from attack by the polishing solution by coating with a lacquer. The polishing bath

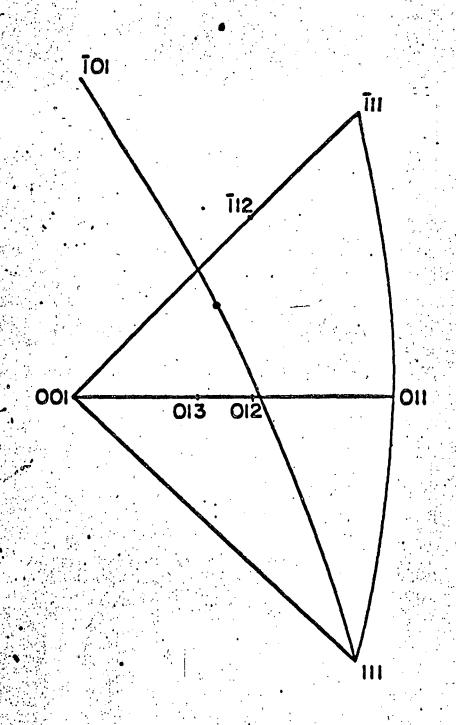


FIG. I. ORIENTATION OF THE TENSILE AXIS OF THE CRYSTAL.

was immersed in ice.

The electropolishing was continued till a layer of material 200 microns in thickness was dissolved away. This was sufficient to remove all traces of mechanical damage induced by machining, as evidenced by the fact that back reflection laue photographs taken subsequently were completely free from asterism.

# Determination of Purity

Specific resistivity measurements were carried out at 273°K, 77°K and  $4.2^{\circ}$ K as a test of purity of the zone melted crystals. It has been shown by Van Torne that a resistivity ratio  $\rho 273/\rho_{4.2}$  of 10,000 in Ta corresponds to a residual level of interstitials of 3 impurity atoms in  $10^9$  matrix atoms. A resistivity ratio of 50,000 was obtained in this case, testifying to the high purity of the crystals.

The experimental set-up used to measure resistivity was essentially the same as that employed by Van Torne. 11 The D.C. current was supplied by a 30 amp Regatron solid state power supply, operated in the constant current mode. An external resistor [0.01 ohm, 100 amps] was included in the circuit to operate the power supply in the constant current mode. This resistor served the additional purpose of measuring accurately the current flowing through the circuit, by knowing the potential developed across it. A millivolt potentiometer was used to measure the potential across the resistor.

A nanovoltmeter was used to measure the potential drop across a known length of the crystal. There was some drift in the nanovoltmeter

reading at the very low potentials developed at liquid helium temperature.

The measurement of resistivity at liquid helium temperature was complicated by the fact that Ta becomes superconducting at 4.38°K. In order to make the resistivity measurement, it was found necessary to increase the current to a value exceeding the superconducting critical current density. For the crystals cross section used, an increase to 10.5 amps was found to be sufficient.

The resistivity measurement at 77°K served to pre-cool the fixture for the measurement at 4.2°K, and reduced the consumption of liquid nelium.

### Flow Stress Measurements

Tensile tests were made with an Instron testing machine, at two strain rates of 9.6 x 10<sup>-5</sup>/sec and 8.05 x 10<sup>-3</sup>/sec. All the specimens were initially prestrained at 550°K. (This is discussed later.) Tests below room temperature were carried out by immersion of the specimen in various constant temperature baths. Tests above room temperature were conducted in a silicone oil bath, with an accuracy of temperature control of ±1°C. The stresses reported are accurate to ±3 x 10<sup>6</sup> dynes/cm<sup>2</sup>.

#### EXPERIMENTAL RESULTS

# Effect of Temperature and Strain Rate on Flow Stress

In order to minimize the scatter from crystal to crystal, each specimen was prestrained at 550°K and a strain rate of 9.6 x 10<sup>-5</sup>/sec to a stress level of about 1% above the yield stress. This was assumed to bring all the crystals to a standard state with about the same level of dislocation density.

An alternate method to achieve a uniform dislocation density would be to anneal the crystals for a fixed time. This is not very practicable in this case, due to the high temperatures and long times necessary.

There is also the likelihood of impurities being picked up during the process.

Figure 2 shows the experimentally determined relationship between the flow stress and temperature for two different strain rates differing by a factor of about 100. It was found that twinning occurred with audible clicks at 77°K at the higher strain rate, and at 4.2°K at the lower strain rate.

All crystals fractured with a chisel edge. At 300°K, there was a total elongation of 24% before fracture, and this decreased to 14% at 77°K. The yield point phenomenon was not observed in any of the crystals that were tested in contrast to the observations of Ferris et al. 12

The critical resolved shear stress for flow in the (101) [111] slip system was calculated using the formula given in Appendix A. The data are presented in Fig. 2. It is seen that the ratio of the flow stress

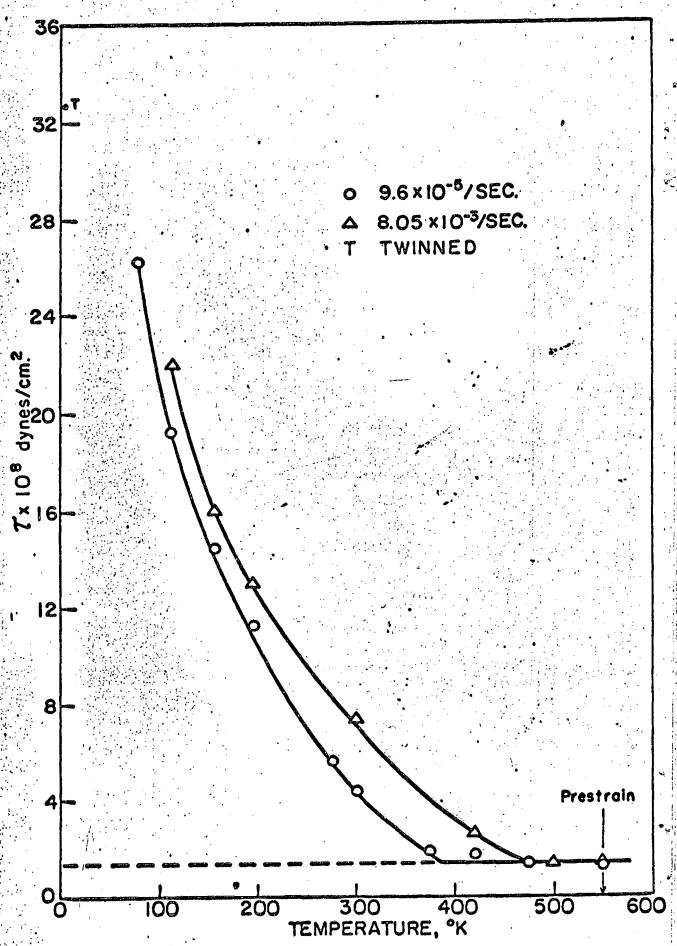


FIG. 2. CRITICAL RESOLVED SHEAR STRESS FOR FLOW vs. TEMPERATURE.

at 77°K to that at 300°K is about 6 to 1.

In the macroscopic deformation of b.c.c. metals, it is known that the flow stress  $\tau$  can be considered to consist of three components.

$$\tau = \tau^* (T, \dot{\gamma}) + \tau a + Kd^n$$
 (1)

 $\tau^*$  is the thermal component dependent on the temperature T, and the strain rate  $\dot{\gamma}$ , which aids in the thermal activation of the rate controlling mechanism;  $\tau a$  represents the athermal component of the stress that must be supplied to push the dislocations over those barriers that cannot be surmounted by thermal fluctuations, such as long range back stresses, attractive junctions etc. In general,  $\tau^*$  decreases sharply with increase in temperature up to the critical temperature but  $\tau a$  is independent of strain rate, and decreases slowly with increasing temperature, in a manner parallel to the variation of the shear modulus G with temperature.

Kd<sup>n</sup> is the component representing the grain size effect. This does not enter into the picture in the present case, since single crystals are under study.

In order to study the effect of temperature on the thermal component of the stress  $\tau^*$ , it has to be separated from others. This can be done by subtracting the stress at a given temperature T from that at the temperature, at or above which  $\tau^*$  becomes zero. From Fig. 2, this temperature is greater than  $400^{\circ}$ K for a strain rate of  $9.6 \times 10^{-5}/\text{sec}$ .

$$\tau^* = \tau_T - (\tau_{550} - \tau_{550}^*)(\frac{G(T)}{G(550)})$$

For the strain rates used,  $\tau_{550}^*$  is practically zero. Hence

$$\tau^* = (\tau_{\rm T} - \tau_{550}) \left( \frac{G({\rm T})}{G(550)} \right) \tag{2}$$

where  $\tau_T$  is the total resolved shear stress for flow at temperature  $T^oK$ , and G(T) is the shear modulus at that temperature. The quantity  $\tau 550 \times \frac{G(T)}{G(550)}$  is then the total back stress, corrected for variation of shear modulus with temperature. The values of  $\frac{G(T)}{G(550)}$  were calculated from Fig. 4, which was obtained from the data of Featherstone and Neighbours. 13 G was taken as the elastic modulus for shear on {110} planes in [111] directions.

$$G = \frac{1}{3} (C_{11} - C_{12} + C_{44}).$$

The values of  $\tau^*$  which are now corrected for specimen variation in  $\tau a$ , are plotted against temperature in Fig. 3 for the two strain rates. The flow stress increases with both decreasing temperature and increasing strain rate. This strongly indicates that the operative deformation mechanism is thermally activated. It can be noticed from Fig. 3 that there is a critical temperature Tc for each strain rate, at which the thermally activated process changes into an athermal process. The critical temperatures are found to be 390°K (\*15°K), Tc<sub>1</sub>, and 470°K (\*15°K), Tc<sub>2</sub> for the lower and higher strain values respectively.

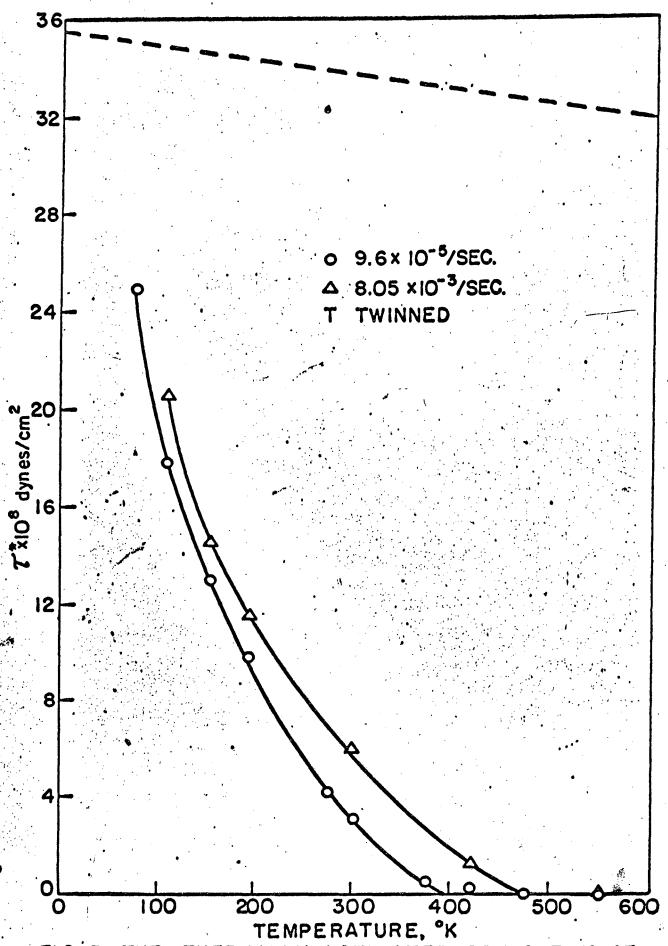


FIG. 3. THE THERMALLY ACTIVATED COMPONENT OF THE FLOW STRESS T s. TEMPERATURE.

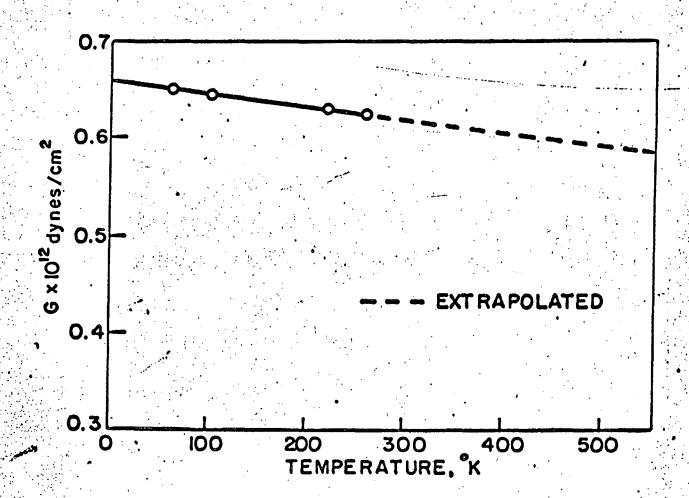


FIG. 4. VARIATION IN SHEAR MODULUS G IN THE (101) [111] SYSTEM WITH TEMPERATURE.

#### **DISCUSSION**

Conrad, and more recently Christian and Masters, have made exhaustive reviews of the available experimental data on the flow stress of the b.c.c. transition metals at low temperatures [<0.25 Tm]. They also examined the validity of the many theoretical models put forward to account for the magnitude of the low temperature stress, and arrived at the conclusion that the resistance of the lattice frictional forces [the Peierls'-Nabarro force] was probably the most important factor in the deformation of the b.c.c. transition metals. When this resistance is appreciable, it is assumed that deformation is controlled by the thermally activated nucleation of kink pairs in straight dislocation lines.

Several approaches to an analytical formulation of the Peierls' mechanism have been attempted, none of which is valid over all stress levels. Recently, Dorn and Rajnak have estimated the saddle point free energy required to nucleate a pair of kinks as a function of applied stress. They have also estimated the effects of stress and temperature on the macroscopic plastic strain rate, when the deformation is controlled by the Peierls' mechanism in terms of the energy required to nucleate a pair of kinks. It will be shown that the present experimental results can be correlated to the theoretical deductions of the Dorn-Rajnak model of nucleation of kink pairs.

The first step in the analysis is to arrive at an accurate estimate of the Peierls' stress at absolute zero  $\tau_p^o$ . This is defined as the maximum shear stress necessary to promote forward motion of the

dislocation from one Peierls' valley to the next, at absolute zero. The lack of experimental data below 77°K, due to the onset of twinning is a handicap in making this estimate. Hence, extrapolation methods have to be resorted to, which inevitably introduce some uncertainty. The best possible value of the Peierls' stress arrived at, by careful extrapolation of the curve (as shown in Fig. 11) between  $\tau^*$  and  $T/T_c$  where  $T_c$  is the critical temperature) is 35.5 x 108 dynes/cm². The error involved in this extrapolation [due to assuming a linear relationship between  $\tau^*$  and  $T/T_c$  at low temperatures] is not more than  ${}^{\pm}2.5 \times 10^8$  dynes/cm².

Having thus settled on a reasonable value of the Peierls' stress, the experimental results can now be quantitatively compared with Dorn and Rajnak's theory. The plastic shear strain rate for the simplest case where the deformation is controlled by the nucleation of pairs of kinks is the geometrically defined length L of dislocation is given by 7

$$\dot{\gamma} = \rho abv \frac{L}{w} e^{-\left(\frac{U_n}{kT}\right)}$$
(3A)

$$= \rho abv \frac{L}{W} e^{\frac{(2U_k - v\tau^*)}{kT}}$$
(3B)

where ρ = density of mobile dislocations

a = distance between Peierls' valleys (about the same order as
the lattice spacing)

b = Burgers' vector

v = Debye frequency

U = saddle point free energy for the nucleation of a pair of kinks
w = width of a pair of kinks at the saddle point free energy
configuration

k = Boltzmann constant

T = temperature in °K

v = activation volume

 $2U_k$  = activation energy to nucleate a kink pair

 $v\tau^*$  = the energy supplied by thermal fluctuations

At T =  $T_c$ , where  $\tau^*$  first becomes zero, the thermal energy that need be supplied is just  $U_n = 2U_k$ , and therefore for this condition,

$$\dot{\gamma} = \rho ab_{\nu} \frac{L}{\nu} e^{-\left(\frac{2U_{k}}{kT_{c}}\right)}$$
(4)

From Eqs. (3A) and (4), assuming that  $\rho$  is independent of temperature,

$$\frac{U_n}{2U_k} \approx \frac{T}{T_c} \tag{5}$$

to a very good approximation.

The theory gives  $U_n/2U_k$  as a function of  $\tau^*/\tau_p$  for different values of  $\alpha$  where  $\alpha$  expresses the deviation of the shape of the Peierls' hill from a purely sinusoidal variation. The shape of the Peierls' hill is defined in terms of  $\alpha$  by

$$\Gamma_{(y)} = \frac{\Gamma_c + \Gamma_o}{2} + \frac{\Gamma_c + \Gamma_o}{2} \left[ \frac{\alpha}{2} + \cos \frac{2\pi y}{a^2} - \frac{\alpha}{4} \cos \frac{4\pi y}{a} \right] \tag{6}$$

 $\Gamma_c$  = line energy of a dislocation at the top of the Peierls' hill where

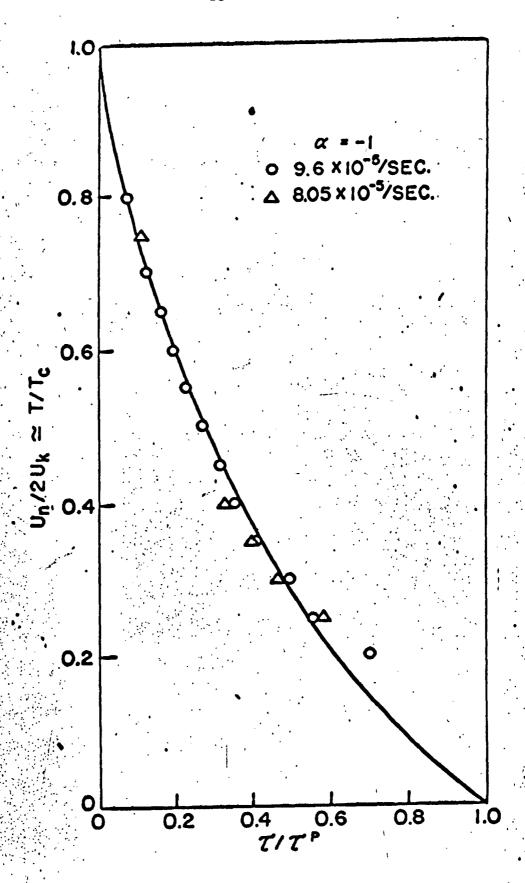


FIG. 5. THE THERMALLY ACTIVATED FLOW STRESS vs. TEMPERATURE IN DIMENSIONLESS UNITS.

y = 0.  $\Gamma_0 = 1$  in energy in the valley where  $y = \frac{8}{2} \cdot \Gamma_{(y)} = 1$  in energy when the dislocation is displaced a distance 'y' from the hill.

It is most significant that the  $U_n/2U_k$  vs.  $\tau^*/\tau_p$  are universal relationships that depend on well defined physical quantities arising from the atomic bonding in the crystal. When  $\tau^*=0$  (at  $T=T_c$ ) the energy to nucleate a pair of kinks is just  $2U_k$  (i.e. twice the kink energy). When the stress equals the Peierls' stress, no additional thermal energy is needed to nucleate kink pairs, since the applied stress is itself capable of doing this, without the aid of thermal fluctuations.

It is now an accepted assumption that the Peierls' stress varies with temperature only through variation in the shear modulus, and this can be represented as

$$\tau_{\mathbf{p}} = \tau_{\mathbf{p}}^{\mathbf{o}} \frac{G(\mathbf{T})}{G(\mathbf{0})} \tag{7}$$

where  $\tau_p^o$  = Peierls' stress at absolute zero, G(T) = shear modulus at temperature T. The variation in the Peierls' stress is shown by the broken line at the top of Fig. 3.

The experimental data of  $\tau^*$  vs. temperature have been replotted in Fig. 5 in terms of  $\tau^*/\tau_p$  vs.  $\frac{T}{T_c}$ , which has been shown equal to  $\frac{U_n}{2U_k}$  to a very good approximation. It can be seen that the experimental points are in good agreement with the theoretical curve representing  $\alpha = -1$ . The only point not coinciding with the curve is that corresponding to  $77^{\circ}$ K. There does not seem to be any specific reason for this, but it is possible that a slight amount of twinning occurred which could have

had the effect of raising the flow stress. However, metallography did not reveal the presence of any twins nor were there any serrations on the load-deflection curve. An alternate explanation could be that the shape of the Peierls' hill is slightly modified at low temperatures [i.e., it may be different from the three values of a shown in the figure].

#### ACTIVATION ENERGY FOR NUCLEATION OF PAIRS OF KINKS

The activation energy can be determined if the change in strain rate due to change in temperature at constant stress is known from Eq. (4). The pre-exponential term  $\rho ab \frac{L}{w} \nu$  may be neglected without serious error, and this gives

$$\frac{\dot{\gamma}_{2}}{\dot{\gamma}_{1}} = \frac{\exp -\left(\frac{2U_{k}(0)}{kTc_{2}} \cdot \frac{G(Tc_{2})}{G(0)}\right)}{\exp -\left(\frac{2U_{k}(0)}{kTc_{1}} \cdot \frac{G(Tc_{1})}{G(0)}\right)}$$

or

$$2U_{k} = \left(\frac{\frac{kG(O)}{G(Tc_{1})} - \frac{G(Tc_{2})}{Tc_{2}}}{Tc_{2}}\right)^{x} \ln \frac{\dot{\gamma}_{2}}{\dot{\gamma}_{1}}$$
(8)

It will be noticed that  $U_k$  is very sensitive to the difference between the reciprocals of the critical temperatures. Any error in  $T_c$  is bound to affect the values of  $2U_k$  significantly.

Using  $k = 1.38 \times 10^{-16} \text{ erg/degree}$ 

$$G(0) = 0.6517 \times 10^{12} \text{ dynes/cm}^2$$

$$G(Tc_1) = 0.6065 \times 10^{12} \text{ dynes/cm}^2$$

$$G(Tc_2) = 0.5965 \times 10^{12} \text{ dynes/cm}^2$$

$$\ln \frac{\dot{\gamma}_1}{\dot{\gamma}_2} = \ln (84.5)$$
 Tc<sub>1</sub> = 390°K, Tc<sub>2</sub> = 470°K.

It is found that  $2U_k(0)$ , the activation energy required to nucleate a pair of kinks at absolute zero is equal to 1.38 x  $10^{-12}$  erg or 0.866 ev.

This computation involves an uncertainty of about  $15^{\circ}$ K in the critical temperatures, and hence the limits of accuracy of  $2U_{k}(0)$  are  $\pm 12\%$ .

Thus, a preliminary judgement on the Peierls' mechanism can be made on the basis of the data plotted in Fig. 5. The most reliable verification of the Peierls' process, however, is the agreement between the theoretical and experimentally deduced activation volumes. The activation volume is defined as the negative of the derivative of the free energy with stress

$$\mathbf{v} = -\frac{\partial \mathbf{U}_{\mathbf{n}}}{\partial \tau^{*}} = - \partial(2\mathbf{U}_{\mathbf{k}} - \mathbf{v}_{\tau^{*}})/\partial \tau^{*}$$
 (9)

The activation volume so defined can be used to eliminate mechanisms with a large value of v. The Peierls' process has quite small activation volumes ranging from 5 to 50 b<sup>3</sup>. It is therefore rather a critical quantity that serves to distinguish the Peierls' process from many other dislocation mechanisms which might on occasions have almost the same activation energy.

Usually, v is not defined directly from Eq. (9), but from the work done by the stress field during the activation. The experimental activation volumes are obtained by the effect of small changes in the strain rate on the flow stress. We define this quantity  $\beta$  as

$$\beta = \frac{\partial \ln \hat{\gamma}}{\partial \tau^*} = \frac{\partial \ln \rho}{\partial \tau^*} - \frac{\partial \ln \omega}{\partial \tau^*} - \frac{1}{kT} \frac{\partial U_n}{\partial \tau^*}$$
 (10)

We take fkT as the apparent activation volume Va, where

$$Va = \beta kT = kT \frac{\partial ln\rho}{\partial \tau^*} - kT \frac{\partial ln\omega}{\partial \tau^*} - \frac{\partial U}{\partial \tau^*}$$
 (11)

where the last term of Eq. (11) is the theoretical activation volume V. Whereas the term containing  $\omega$  is always negligibly small, Va can on occasions be slightly larger than V, as a result of the possible increase in dislocation density  $\rho$ , as the stress is increased. The apparent activation volumes deduced in this manner from the data in Fig. 3, are plotted against  $\tau^*$  in Fig. 6. It can be seen that at the high stress levels (above about  $^{14}$ .5 x  $10^8$  dynes/cm<sup>2</sup>), the activation volume is constant at about 25 b<sup>3</sup> (where b is the Burgers' vector). This low value establishes the Peierls' mechanism as the operative one.

Figure 8 shows the theoretical plot of the activation volume here as the thermally activated component of the flow stress in dimensionless units for different values of  $\alpha$ . The experimental values of  $(\tau_p/2U_k)\ V_a$  are also plotted in the same figure. It is not possible to fit the data to any one particular curve, owing to the scatter in the results. Within the limits of experimental error, the agreement with theory is satisfactory.

The activation energy is determined from the equation

$$U_{n} = \frac{\Delta \ell_{n} \dot{\gamma}}{\Delta (-\frac{1}{kT})}$$

This is plotted in dimensionless units versus  $\tau^*$  in Fig. 7. The theoretical curve is also shown in the same figure. There may be considerable error involved in computing  $U_n$ , since it depends on differences between reciprocals of temperature, which are read off from the  $\tau^*$  vs. T curves. In view of this, scatter in the values is inevitable, and the data are in broad agreement with Dorn and Rajnak's theory.

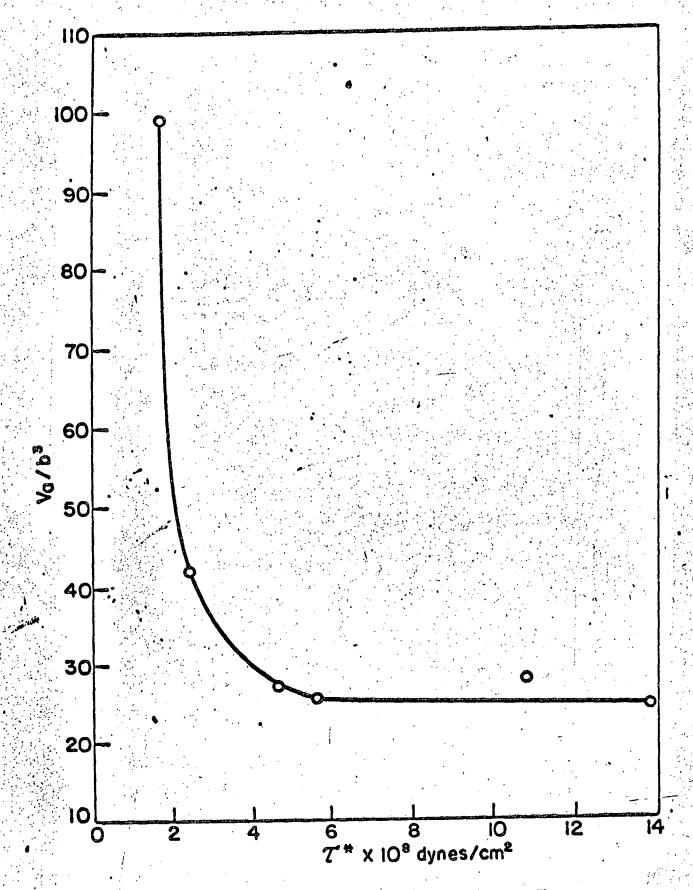


FIG. 6. APPARENT ACTIVATION VOLUME VS.
THERMALLY ACTIVATED COMPONENT OF THE
FLOW STRESS.

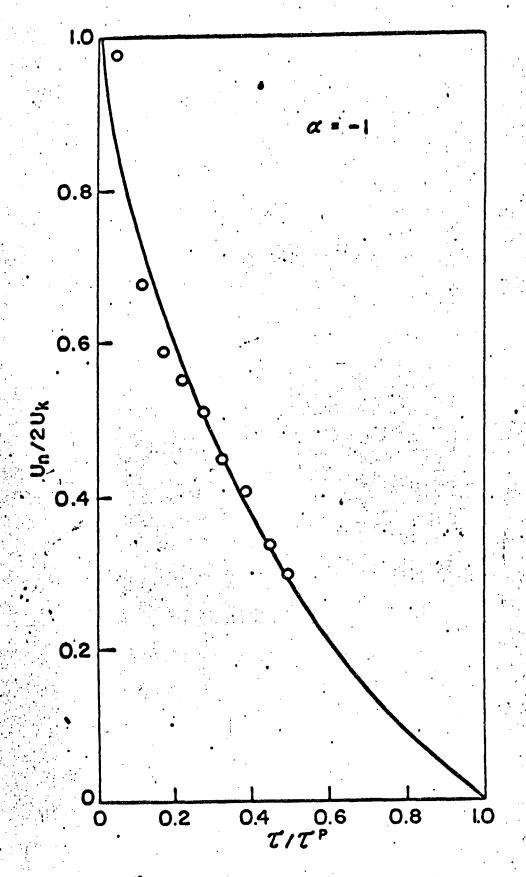


FIG. 7. THE THERMALLY ACTIVATED FLOW STRESS vs. ACTIVATION ENERGY IN DIMENSIONLESS UNITS.

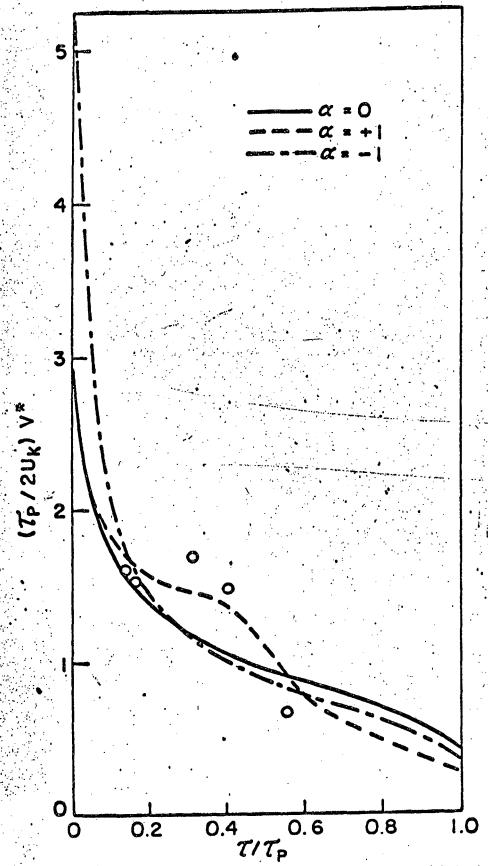


FIG. 8. THE THERMALLY ACTIVATED FLOW STRESS vs. ACTIVATION VOLUME IN DIMENSIONLESS UNITS.

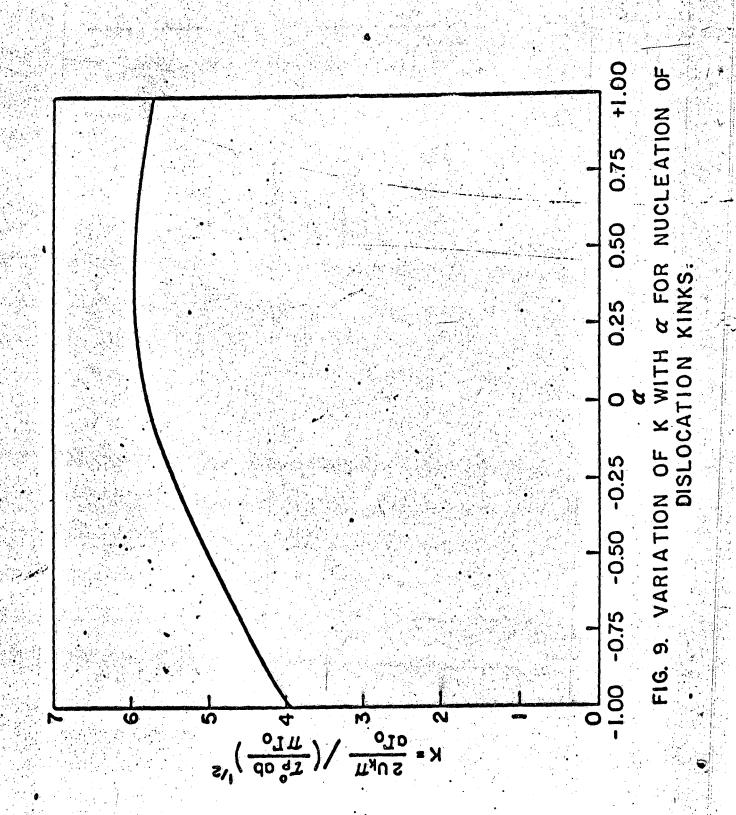
# EVALUATION OF LINE TENSION OF DISLOCATION SITUATED IN THE PEIERLS' VALLEY

To evaluate the line tension, we make use of a relationship given by Dorn and Mitchell, for the kink energy

$$K = \frac{2U_{k}\pi}{a\Gamma_{o}} \left| \left( \frac{T_{\rho ab}^{o}}{\pi\Gamma_{o}} \right)^{1/2} \right|$$

In the above expression,  $2U_k$  and  $\tau^o$  have been determined experimentally, and appropriate values for a and b can be substituted. The variation of K with  $\alpha$  has been determined by Dorn and Mitchell, and this is shown in Fig. 9. Thus,  $\Gamma_o$  is the only unknown quantity, and hence can be calculated. Since the experimental plot of  $\frac{U_n}{2U_k} = \frac{T}{T_c}$  versus  $\tau^*/\tau^o_\rho$  as shown in Fig. 5 seems to fit best with the curve for  $\alpha = -1$ , we take the value of K = 3.9 corresponding to  $\alpha = -1$ . The calculation gives  $\Gamma_o = 10.65 \times 10^{-4}$  dynes, which is approximately equal to 2 Gb<sup>2</sup>. This is not too unreasonable, in view of the 12% error in  $2U_k$ , and the 7% in the Peierls' stress.

Figure 10 shows the variation of the activation volume as a function of strain at 273°K. Whereas the activation volume should decrease with increase in strain, if the intersection mechanism were operative, this is not the case. It remains constant with strain as suggested by the Peierls' mechanism.



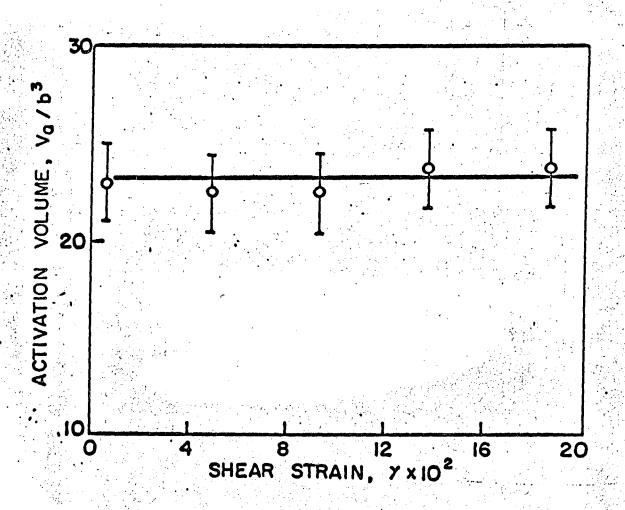


FIG. 10. ACTIVATION VOLUME AS A FUNCTION OF STRAIN.



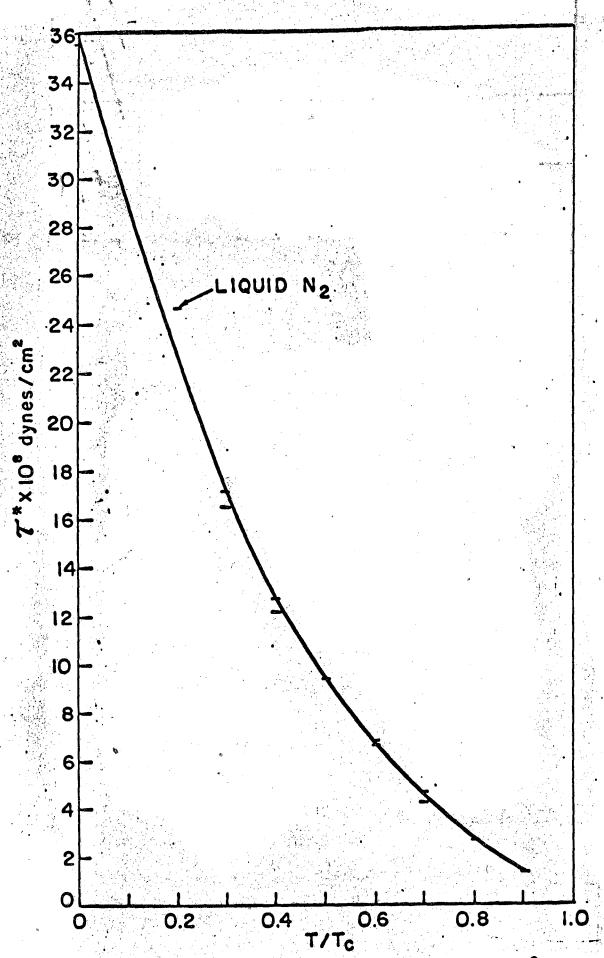


FIG. II. EXTRAPOLATION TO DETERMINE To

#### CONCLUSIONS

The deformation of single crystals of high purity tantalum has been investigated in the low temperature range, at two strain rates.

The following conclusions can be drawn.

- (1) The relation of stress vs. temperature, activation energy vs. stress and activation volume vs. stress agree very well with the Peierls' process when the rate controlling mechanism is by nucleation of pairs of kinks.
- (2) The activation energy of the process of the nucleation of pairs of kinks is estimated to be 0.86 eV, to within 12%.
- (3) The activation volume is about 25 b<sup>3</sup> (to within 10%), except at very small stresses.
- The estimated value of the dislocation line tension in the Peierls' valley is  $\Gamma_0 = 10.65 \times 10^{-4}$  dynes. This is an approximate value, but it is within the right order of magnitude, and could be said to be reasonable.

#### APPENDIX A

The critical resolved shear stress for flow and the shear strain were computed using the formulae given below.

$$\tau = \frac{L}{A_o} \sin x_o \sqrt{1 - \left(\frac{\ell_o}{\ell}\right)^2 \sin^2 \lambda_o}$$
 (A-1)

$$\gamma = \frac{\sqrt{(\ell/\ell_0)^2 - \sin^2 \lambda_0 - \cos \lambda_0}}{\sin x_0}$$
 (A-2)

where :

L = instantaneous load

 $A_{o}$  = initial cross sectional area of the specimen

lo = initial length of the specimen

l = instantaneous length

 $\lambda o$  and  $x_o$  are the initial Schmid angles.

The above equations apply for single slip.

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#### EFERENCES

- 1. H. Conrad, "Yielding and Flow of the B.C.C. Metals at Low Temperatures"

  Presented at the Symposium on the Relation Between Structure and

  Properties of Metals, National Physical Laboratory, Teddington,

  Middlesex, England, January, 1963.
- 2. J. W. Christian and B. C. Masters, "Low Temperature Deformation of Body Centered Cubic Metals", Proc. Roy. Soc., <u>A281</u>, 223, (1964).
- 3. T. E. Tietz and J. W. Wilson, "Behavior and Properties of Refractory Metals", Stanford University Press, Stanford, California, 1965, p223.
- 4. T. E. Mitchell and W. A. Spitzig, "Three Stage Hardening in Tantalum Single Crystals", Acta Met., 13, 1169, (1965)
- 5. A. Seeger, "On the Theory of the Low Temperature Internal Friction Peak Observed in Metals", Phil. Mag., 1, 651, (1956).
- 6. J. Lothe and J. P. Hirth, "Dislocation Dynamics at Low Temperatures",
  Phys. Rev., 115, 543, (1959).
- 7. J. E. Dorn and S. Rajnak, "Nucleation of Kink Pairs and the Peierls' Mechanism of Plastic Deformation", Trans. Met. Soc. AIME, 230, 1052, (1964).
- 8. A. K. Mukherjee and J. E. Dorn, The Rate Controlling Mechanism for Slip in the Intermetallic Compound AgMg at Low Temperatures", Trans. Met. Soc. AIME, 230, 1065, (1964).
- 9. A. Rosen, J. D. Mote and J. E. Dorn, "Mechanism for Thermally Activated Prismatic Slip in Ag<sub>2</sub>Al", Trans. Met. Soc. AIME, <u>230</u>, 1070, (1964).
- 10. P. Wynblatt, A. Rosen and J. E. Dorn, "The Effect of Temperature,

- Strain Rate and Structure on the Flow Stress of an Fe-2% Mn Alloy", Trans. Met. Soc. AIME, 233, 651, (1965).
- of Tantalum-Molybdenum Single Crystals", Ph.D. Thesis, University of California, Berkeley, January, 1965. To be published in J. Less Common Metals.
- 12. D. P. Ferris, R. M. Rose and J. Wulff, "Deformation of Tantalum Single Crystals", Trans. Met. Soc. AIME, 224, 975, (1962).
- 13. F. H. Featherstone and J. R. Neighbours, "Elastic Constants of Ta, W and Mo", Phys. Rev., 130, 1324, (1963).
- 14. J. E. Dorn and J. B. Mitchell, "Slip Mechanisms in Single Crystals of H.C.P. Phases", Proceedings after Second Berkeley International Materials Conference, June, 1964. Edited by V. F. Zackay, John Wiley and Sons, Inc., New York, 1965.

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