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FUNDAMENTAL STUDIES RELATED TO THE ORIGIN AND NATURE OF CREEP OF METALS

Seventh Technical Report

Stress Induced Movement of Crystal Boundaries

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INTRODUCTION

Experimental observations in three fields, namely those of crystal growth, strength of small metal crystals, and grain boundary energies have helped establish the soundness of dislocation theory. Recently added to this list is the observation of stress induced motion of small angle boundaries. The possibility that a small angle boundary might move under the action of a suitable system of shear stresses was suggested by Burgers[1] and developed more fully by Shockley[2]. The predicted motion of small angle boundaries under stress was first observed by Washburn and Parker[3]. Experimental evidence for this boundary movement was obtained through observations of a 2° boundary in a zinc crystal loaded as a cantilever beam. In the interim, a number of additional observations of boundary motion have been made; these are reported herein.

EXPERIMENTAL PROCEDURES AND OBSERVATIONS

A. Single Small Angle Grain Boundaries

The crystals used in this boundary motion study were prepared from 99.99% Horsehead Special zinc by a modified Bridgeman technique. The earlier observations were confined to small angle boundaries formed incidental to solidification or cooling of the crystal. For the present investigation a technique was developed for intentionally introducing a boundary. Spherical crystals were cleaved at -196°C to form disc-shaped specimens approximately one-half inch in thickness. Each specimen was supported as a simple beam and heated to 350°C. The load was applied at the midspan through a knife edge perpendicular to the basal plane, introducing local plastic bending. After cleaving away the upper and lower surfaces, the specimen was annealed for one hour at 400°C. Although it was customary to introduce the boundaries perpendicular to a slip direction in the crystal, this method permitted arbitrary location and orientation of the boundaries, and facilitated studies of their dynamic properties.

Boundary angles were measured with a long focal length reflectograph; the estimated error was ± 4 minutes. The disc-shaped specimen containing the boundary was mounted in U-shaped grips and loaded as a cantilever beam inside a small resistance furnace. Observations were made with an optical microscope looking down on the cleaved surface of the crystal in the manner schematically represented in Fig. 1. Oblique illumination was used so that the location of the grain boundary was indicated by the difference in brightness across the boundary. Displacement of the boundary was measured with a Filar eyepiece which permitted the position of the boundary to be established within ± .001 mm. In several instances, it was deemed desirable to record the boundary movement on motion picture film for further study and analysis. When this was the case, the Filar eyepiece was removed and the body of the camera joined to the microscope with an extension tube. The objective lens of the microscope functioned as the camera lens.

Although the majority of observations to date have been qualitative in nature, a number of quantitative results are included in the following summary of experimental observations:

1. With the crystal loaded as a cantilever beam as indicated in Fig. 1, the boundary was observed to move towards the left under the influence of the shearing stresses. The calculated critical shear stress for the boundary motion was of the order of the magnitude of the critical shear stress for slip in zinc. When the direction of the stress was reversed, the direction of the motion of t' boundary was also reversed and the boundary moved to the right past its original position. A series of pictures selected from the movie are shown in Fig. 2. The time interval between pictures was approximately ten seconds. It should be pointed out that while there are no obvious crystallographic markings on the cleavage face of the specimen, water stains on the surface serve as convenient reference markers for the progress of the boundary movement.

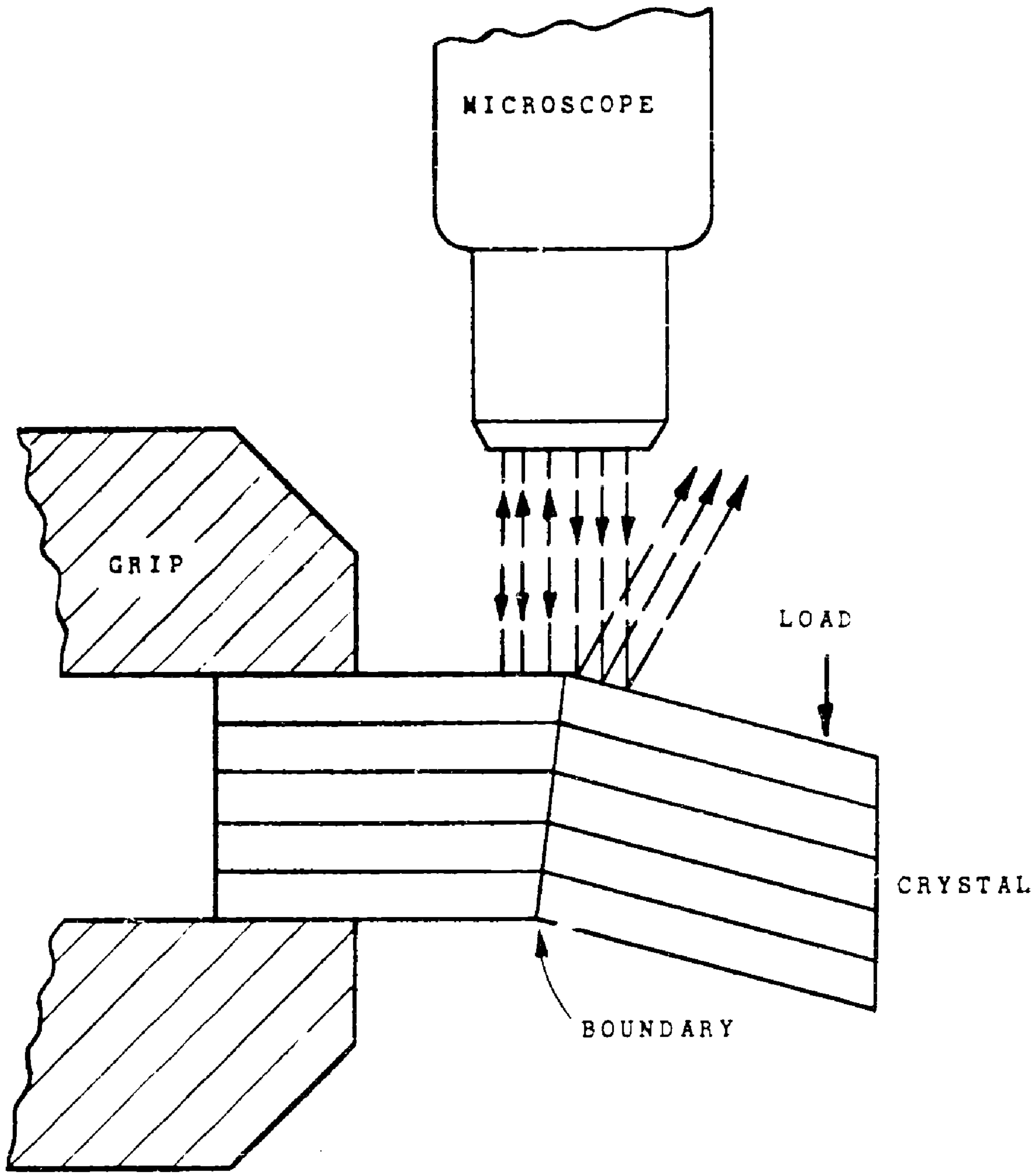

FIG. 1 ILLUSTRATION OF LOADING METHOD FOR BOUNDARY MOVEMENT STUDIES WHICH WOULD MOVE BOUNDARY TO LEFT. HORIZONTAL LINES INDICATE SLIP DIRECTION; SLIP PLANES PERPENDICULAR TO PLANE OF PAPER.

FIG. 2 SERIES OF PHOTOGRAPHS OF A MOVING BOUNDARY SHOWING THAT DIRECTION OF MOTION REVERSED WHEN SHEAR STRESS WAS REVERSED. DIRECTION OF MOTION WAS REVERSED TWICE. 350°C AND 100x.

With another crystal, the load required to move the boundary at a constant rate was measured at room temperature. The results are reproduced in Fig. 3. A significant, but progressively decreasing reduction of the critical load was noted with each reversal of the loading direction.

2. It was the general observation that under a given load, the smaller the boundary angle, the more rapid was the displacment. The sequence of pictures in Fig. 4 records the movement of two boundaries at 350°C. The boundary on the left was approximately one-half degree larger than the one on the right. Under the applied shear stresses, both boundaries moved towards the left, but at unequal rates. The more rapid movement of the smaller boundary resulted in a shortening of the distance between the two boundaries.

3. Whenever one moving boundary overtook another, the two united to form a single boundary. It was found that the new boundary required higher stresses to make it move. A typical load displacement curve illustrating this is shown in Fig. 5. The manner in which the union occurred is recorded in the series of pictures of Fig. 6. Union between the boundaries involved motion of the junction along the larger of the two boundaries. When the external stress was reversed during the process, the direction of motion of the junction was also reversed. The result was a gradual separation of the new boundary into the two original boundaries.

4. The motion of a boundary was retarded in the vicinity of certain imperfect regions in the crystal. The restraint imposed by the distorted material surrounding a Tukon hardness indentation is indicated in Fig. 7. The photographs show that locally the radius of curvature of the boundary steadily decreased as the boundary approached the deformed area.

When the degree of cold work was much less, the movement of the boundary was not appreciably affected. This condition is illustrated in Fig. 8, where the boundary can be observed to pass through the area with little or no retardation.

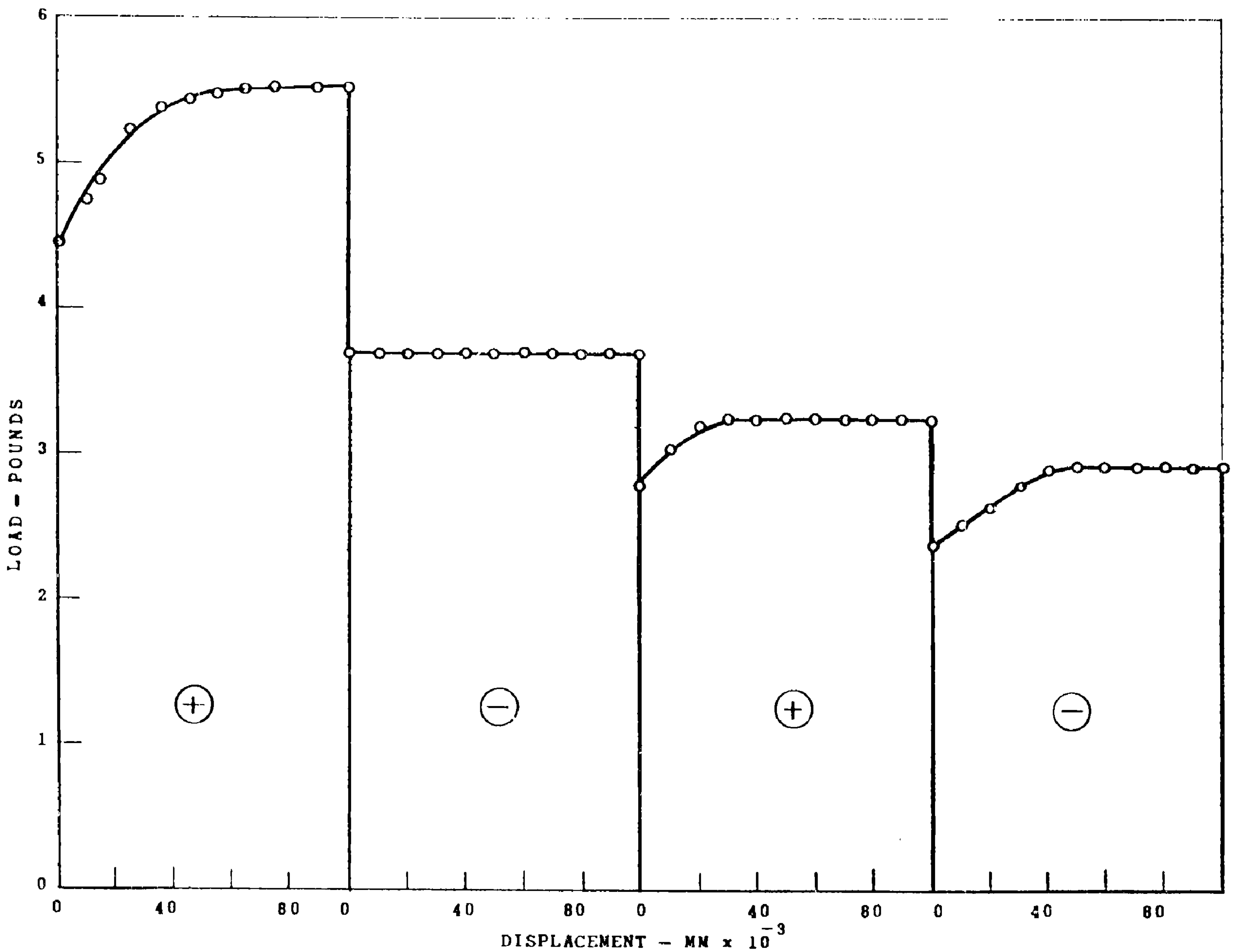

FIG. 3 SERIES OF CURVES FOR A MOVING BOUNDARY SHOWING DECREASE IN HEIGHT OF LOAD-DISPLACEMENT CURVE WHEN DIRECTION OF MOVEMENT WAS REVERSED. RATE OF BOUNDARY MOVEMENT HELD APPROXIMATELY CONSTANT. 25 °C

FIG. 4 ILLUSTRATION SHOWING THAT RATE OF BOUNDARY MOTION DECREASES WITH INCREASING BOUNDARY ANGLE . ONE-HALF DEGREE BOUNDARY ON RIGHT OVERTAKING ONE DEGREE BOUNDARY ON LEFT. 350 °C AND 100x.

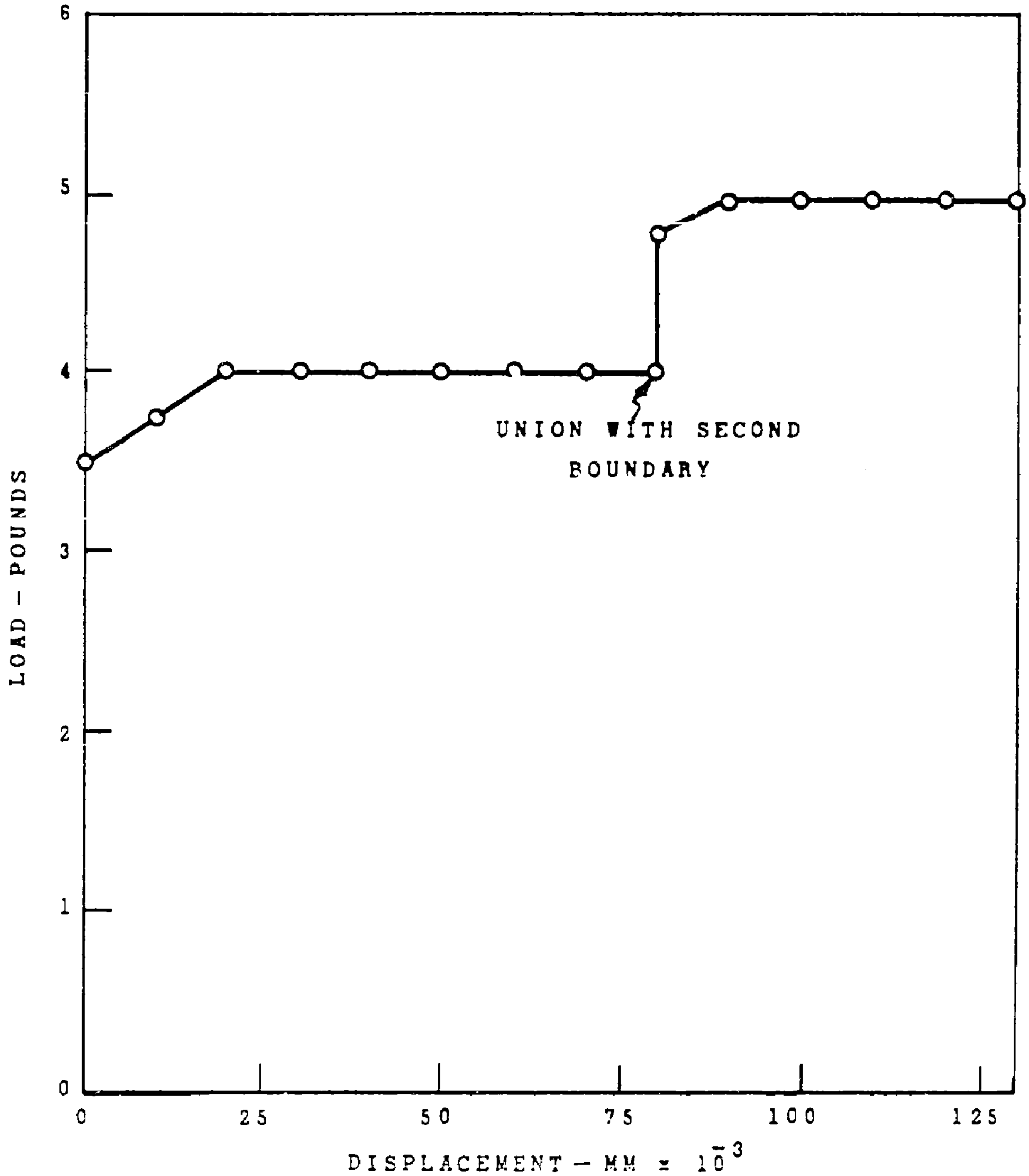

FIG. 5 LOAD-DISPLACEMENT CURVE SHOWING THAT WHEN TWO BOUNDARIES UNITE THE LOAD REQUIRED FOR CONTINUED MOVEMENT IS INCREASED. RATE OF MOTION HELD APPROXIMATELY CONSTANT. 25°C

FIG. 6 PHOTOGRAPHS SHOWING SUCCESSIVE STAGES OF STRESS INDUCED UNION OF TWO SMALL ANGLE BOUNDARIES. 350 °C AND 100x.

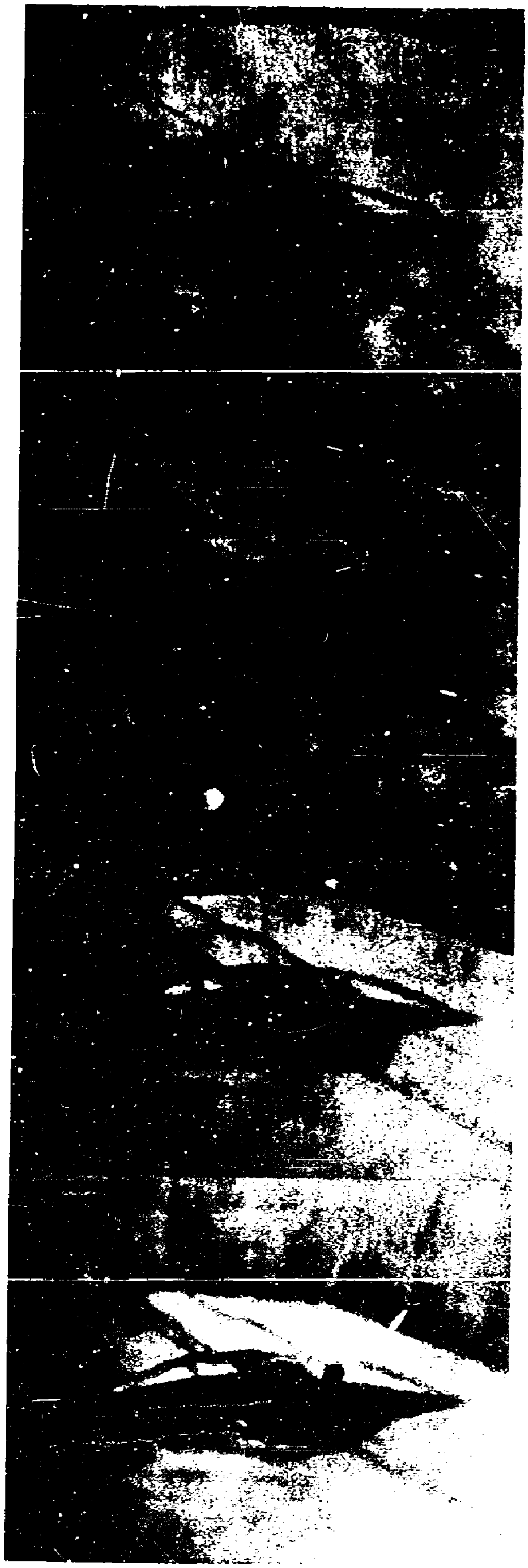

FIG. 7 RETARDATION OF BOUNDARY MOVEMENT BY A HEAVILY DISTORTED REGION. 350°C AND 100x.

FIG. 8 UNHINDERED PASSAGE OF BOUNDARY THROUGH
A LESS SEVERELY DISTORTED REGION.
350 °C AND 100x.

5. As might have been expected, the rate of movement of a boundary under a given load increased as the temperature was raised. A summary of the rates measured in the temperature range of 300 to 400°C at a stress level of approximately 8 psi is represented in Fig. 9.

B. Hexagonal Systems of Small Angle Boundaries

Another form of low angle boundary is that associated with the formation of a hexagonal-shaped substructure in zinc crystals. When a disc-shaped specimen having the basal plane parallel to the surface is heated to 350°C and a concentrated load is applied parallel to the c-axis by means of a conical pointed indenter, a hexagonal system of low angle boundaries is developed. This is shown schematically in Fig. 10. At these temperatures, slip occurs internally on the basal planes. Dislocations introduced initially by the local plastic bending collect in a hexagonal array to form low angle boundaries. Details of the growth of such a substructure under continued loading were best followed with a binocular microscope.

The more important experimental observations pertinent to the movement of hexagonal arrays of low angle boundaries may be summarized as follows:

1. Under conditions of constant load, growth of the substructure eventually stopped. Stages in the growth of the structure at 350°C are illustrated in the series of pictures of Fig. 11. After motion of the system of boundaries had come to a halt, movement was resumed if the load was increased.

2. A second but inactive substructure of this type was a sufficient barrier in the path of an actively enlarging system to distort the growing hexagon. To illustrate this point, a small hexagonal substructure was introduced into the crystal in the manner previously described; it may be seen in the lower right hand corners of the pictures in Fig. 12. The load was immediately removed, and the point of application shifted to the approximate center of the crystal face, where a second substructure was formed and allowed

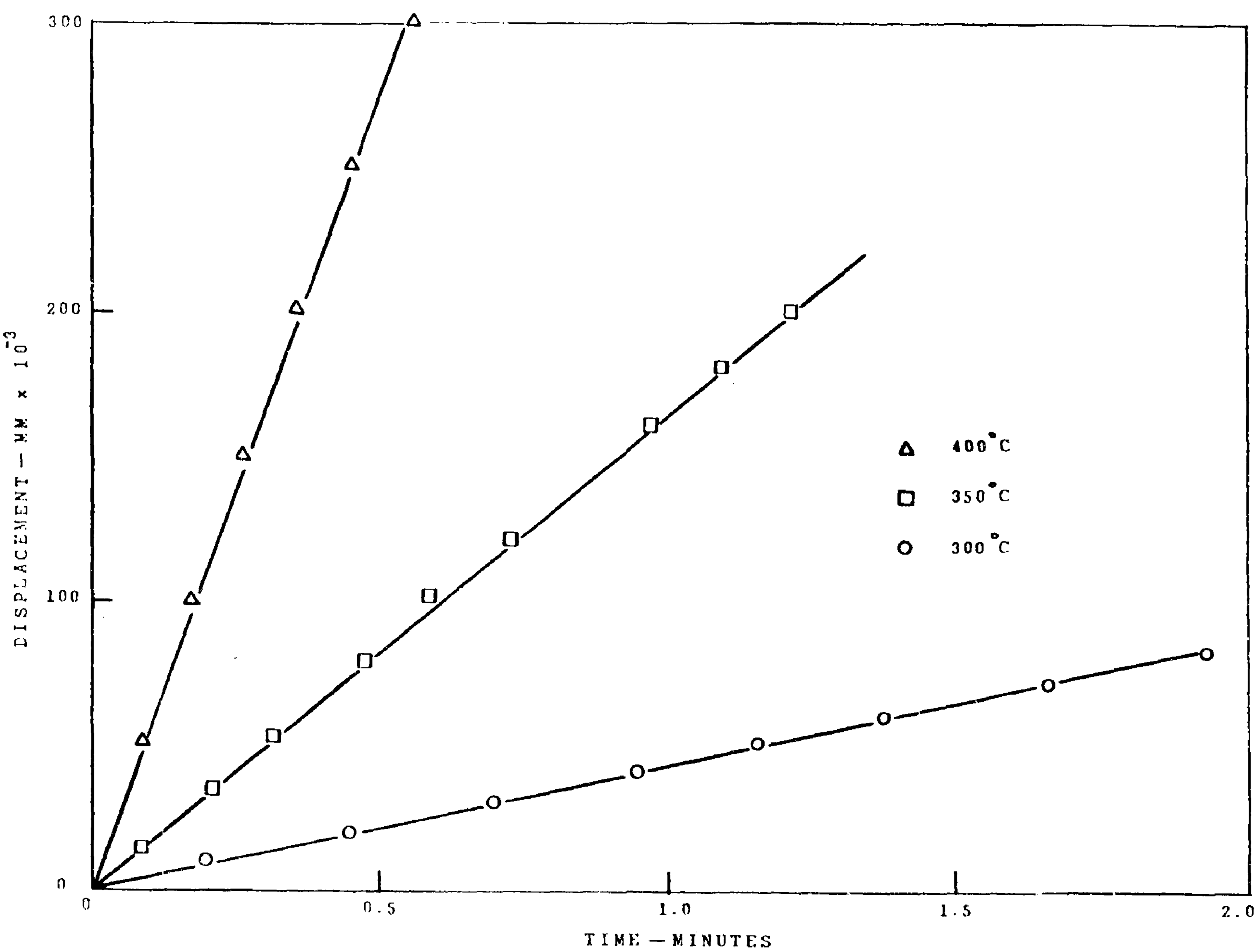

FIG. 9 DISPLACEMENT-TIME CURVES AT VARIOUS TEMPERATURES SHOWING RATE OF MOVEMENT OF A TYPICAL BOUNDARY UNDER CONSTANT LOAD.

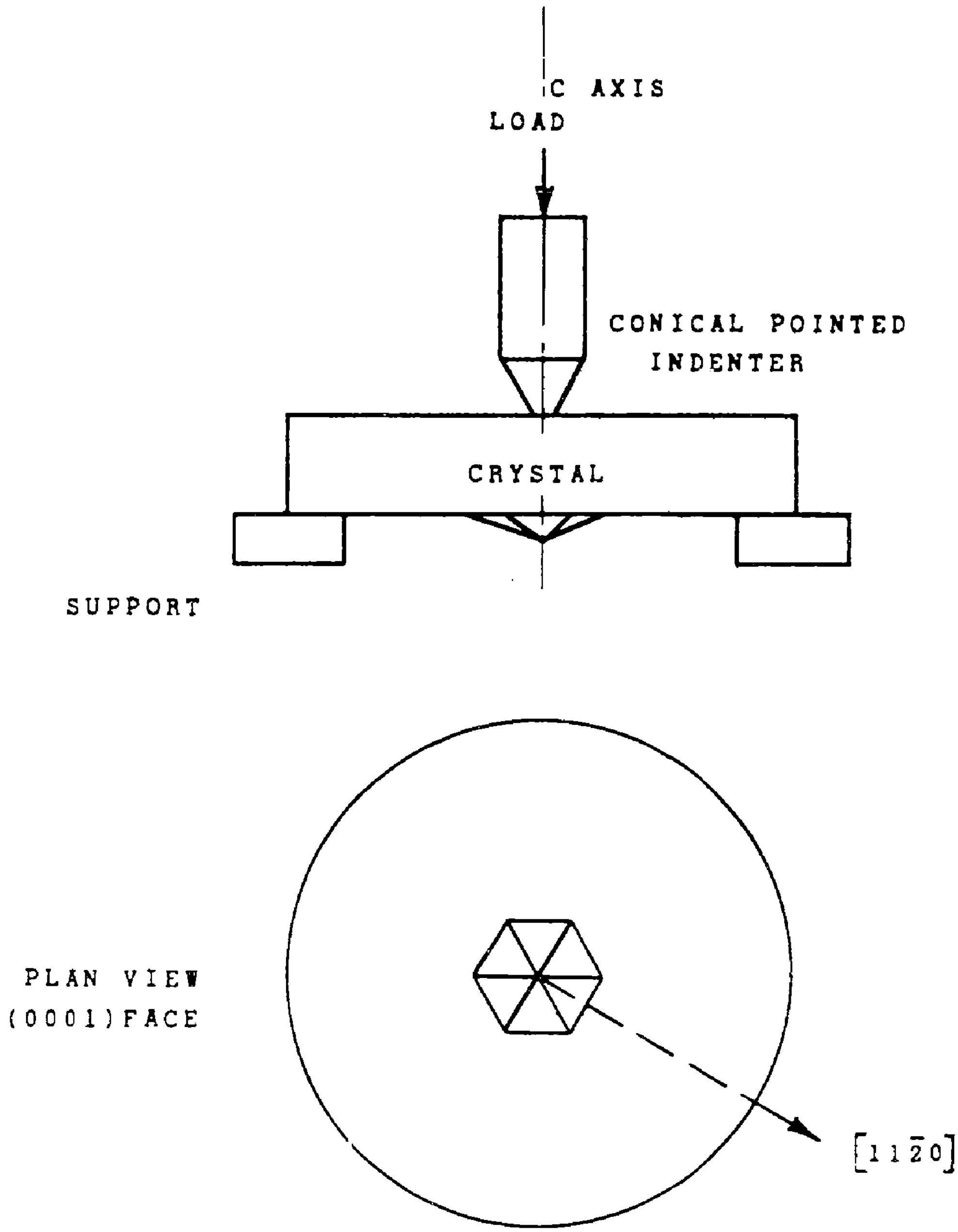

FIG. 10 SKETCH SHOWING METHOD OF INTRODUCING A HEXAGONAL SYSTEM OF LOW ANGLE BOUNDARIES INTO A CRYSTAL.

FIG. 11 STAGES IN THE GROWTH OF A HEXAGONAL SYSTEM OF LOW ANGLE BOUNDARIES.
350 °C AND 10×.

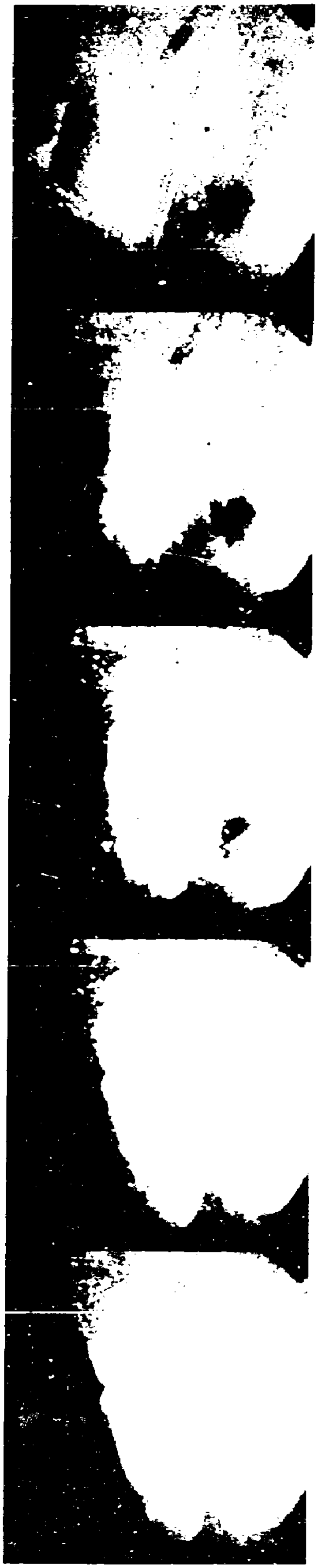

FIG. 12 RETARDATION OF A GROWING HEXAGONAL SYSTEM OF BOUNDARIES IN THE VICINITY OF AN INACTIVE SYSTEM. 350 °C AND 10×.

to spread under constant load. There was a marked reluctance of the boundaries of the active substructure to pass through the previously formed substructure, causing the distortion shown in the last two pictures.

3. To a somewhat lesser extent, a single small angle boundary restricted the movement of the spreading hexagonal substructure. A single straight small angle boundary was introduced into the plate-shaped crystal specimen approximately halfway from the center of the face to the edge. The crystal was then loaded to produce a hexagonal substructure that was allowed to enlarge until its boundaries impinged upon the single straight boundary. The most important feature here, aside from the distortion of the hexagonal substructure, was the repulsion between the single boundary and the hexagon. As may be seen in Fig. 13, from its initial approximately straight condition, the boundary was altered to the extent of becoming noticeably curved.

DISCUSSION

It has become apparent that plastic properties of crystals are largely determined by the presence of imperfections. The nature and distribution of these imperfections, however, and the changes accompanying plastic flow have remained obscure. Single imperfections may never be observed unless magnifications great enough to see individual atoms are achieved. However, simple small angle boundaries represent groups of imperfections whose motion through a crystal can be observed and controlled. Their behavior seems to require that they consist of an array of edge dislocations of like sign and equal Burgers vector distributed more or less uniformly over the plane of the boundary. Whether or not progress leading to a better understanding of plastic flow can be made with moving boundary investigations will depend in large measure upon the development of more satisfactory experimental techniques.

The somewhat limited observations made to date appear to be consistent with other observations of plastic behavior. Within the temperature range

FIG. 13 DEFLECTION OF A SINGLE LOW ANGLE BOUNDARY IN THE PATH OF A GROWING HEXAGONAL SYSTEM. 350 °C AND 10x.

300 to 400°C, the displacement rate, $\dot{\mathcal{E}}$, of the dislocation array through the lattice under a given stress varies with temperature in accordance with the expression:

$$\dot{\mathcal{E}} = Ae^{-Q/RT}$$

where A = a constant

Q = the activation energy

R = the gas constant

T = the absolute temperature

The value of the constant Q has been measured as approximately 21,500 calories per mole (Fig. 14). It is interesting to note that the following temperature dependent processes in zinc crystals give very similar values for the activation energy:

Self diffusion parallel to c-axis	20,400 cal/mol	(4)
Creep	20,000	(5)
Recovery after pure slip deformation	20,000	(6)

The striking agreement of the activation energies of all these processes suggests that all may involve the same basic atomic movements. This fundamental process may be interaction of vacant lattice sites with edge dislocations. The fact that motion of this dislocation array is at a rate dependent on temperature in the same way as self diffusion suggests that dislocations encounter barriers which impede their movement. The capacity of the barrier to block a section of the dislocation line appears to be removed by a diffusion process, possibly by the motion of edge dislocations at right angles to their Burgers vector through interaction with lattice vacancies. The thought that dislocations moving through a lattice encounter barriers is also suggested by the observed lowering of the critical stress when the boundary was moved back and forth through the same volume of crystal. Perhaps with repeated reversals of direction of motion, the boundary dislocations encountering barriers move out

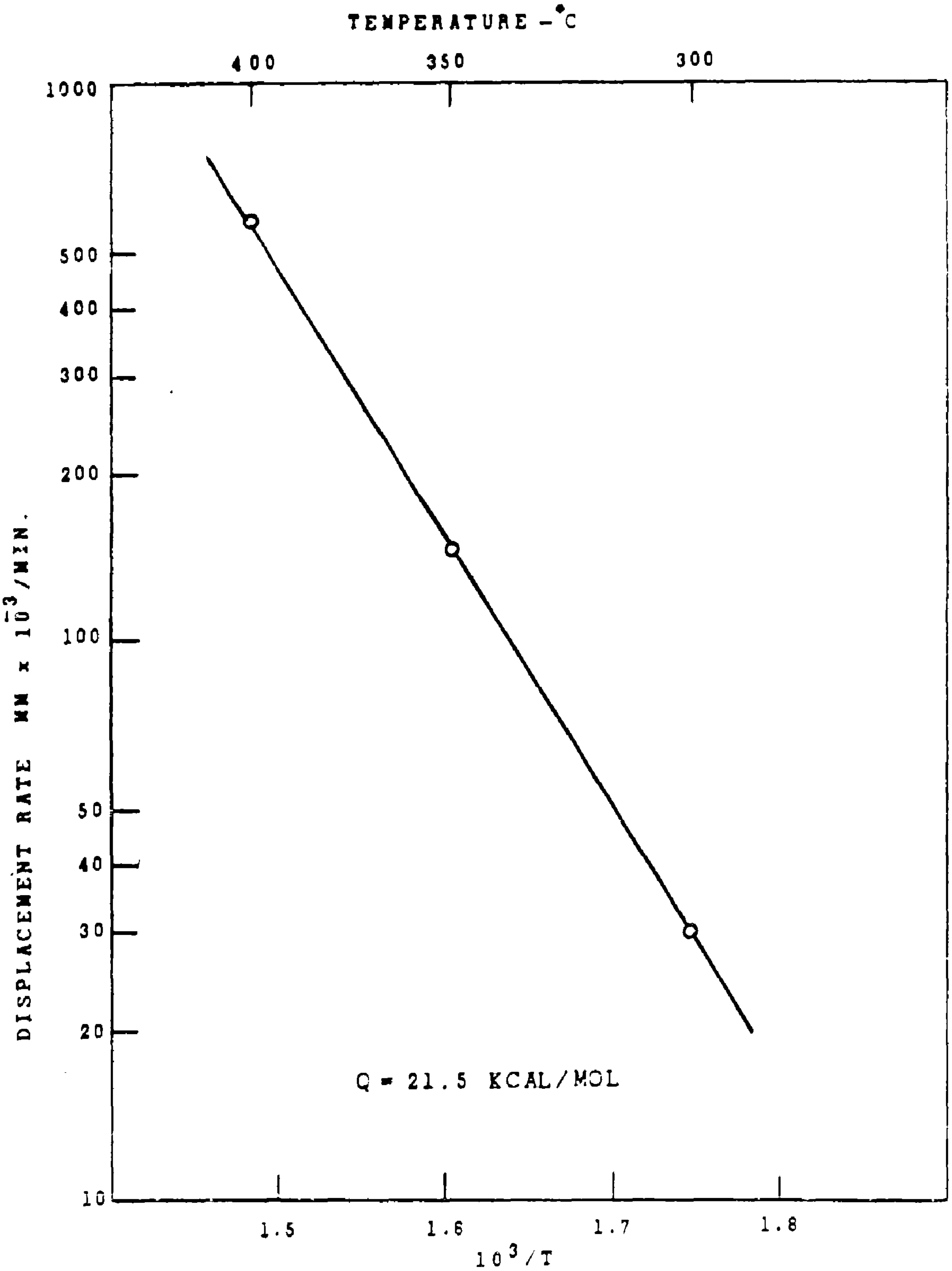

FIG. 14 THE LINEAR RELATIONSHIP BETWEEN LOG DISPLACEMENT RATE AND RECIPROCAL OF THE ABSOLUTE TEMPERATURE YIELDS AN ACTIVATION ENERGY OF APPROXIMATELY 21.5 KCAL / MOL FOR BOUNDARY MOTION AT HIGH TEMPERATURES. STRESS CONSTANT AT 8 P.S.I.

of their original planes into nearby ones where motion is relatively easy. An alternate interpretation of the results is that boundary dislocations become trapped and are removed from the boundary. As a consequence, the boundary angle becomes smaller and the boundary easier to move. Future experiments should clarify this point.

At low temperatures and higher stresses, it is possible to move dislocation arrays under conditions where appreciable diffusion cannot occur. Under these conditions it is possible that experimental verification of Shockley's (7) prediction of a very low activation energy for movement of a dislocation may be obtained.

A mechanism for the formation of a substructure in plastically deformed crystals is suggested by the uniting of small angle boundaries under stress to form larger angle boundaries. With the application of external stresses, it is a reasonable assumption that small angle boundaries in crystals could form and migrate until union was effected with other moving boundaries. Stress induced movement of the boundaries could continue until the angular magnitude of all internal boundaries attained a size characteristic of that temperature and stress level. A stable substructure could thus be created without any special activation energy being required other than that associated with the stress induced motion of the dislocation arrays.

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