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PLASTIC DEFORMATION OF MAGNESIUM OXIDE SINGLE CRYSTALS AT ELEVATED TEMPERATURES

Stephen M. Copley
(Ph. D. Thesis)

April 1, 1964
# PLASTIC DEFORMATION OF MAGNESIUM OXIDE SINGLE CRYSTALS AT ELEVATED TEMPERATURES

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IV. Summary 

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Appendix A 

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PLASTIC DEFORMATION OF MAGNESIUM OXIDE SINGLE CRYSTALS AT ELEVATED TEMPERATURES

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ABSTRACT

Carefully controlled stress-strain measurements have been obtained for MgO single crystals compressed at a constant stress rate, with \(\langle 100\rangle\) and \(\langle 111\rangle\) stress axes, at temperatures ranging from 1000 to 1600°C. These have been correlated with etch pit and stress birefringence studies of the distribution of dislocations in single crystals at various stages of deformation. Similar correlations have also been made in the temperature range 26 to 1000°C, where the stress-strain behavior of MgO has already been fairly well established by previous investigations. A general discussion of the complications which arise in making and interpreting stress-strain measurements in compression is given.

A. \(\langle 100\rangle\) Specimens

Specimens with \(\langle 100\rangle\) stress axes deform plastically by slip on \(\{110\}\) \(\langle 110\rangle\) slip systems. At room temperature, the shape of the stress-strain curve is determined by the dislocation mobility and the number of slip bands formed. Dislocations only move in the undeformed regions at slip band edges. Strain hardening results from slip band overlap.

Between 26 and 1000°C, the dislocation mobility increases with increasing temperature, thus causing the yield stress to decrease. The etch pit density in slip bands formed at 500°C is less than at room temperature, while the total shear strain is about the same. At 1000°C, slip bands still form; however, dislocations were also found to be distributed throughout the volume of the crystal. This effect was attributed to the initiation of slip by grown-in dislocations. The slope
of the stress-strain curve, after yielding, is about five times greater at 1000°C than at room temperature because there are no longer undeformed regions in which dislocations can move.

Above 1100°C, the yield stress decreases slowly with increasing temperature, and the strain rate becomes much less stress sensitive. The strain-hardening rate decreases rapidly owing to the annealing out of thermally unstable dislocation damage such as dislocation dipoles. At 1300°C, polygonization walls, and dislocation boundaries formed by dislocations lying on oblique slip planes, obstruct dislocation movement. Above 1400°C, polygonalization walls were no longer observed to form.

B. (111) Specimens

Specimens with (111) stress axes deform plastically by slip on the {100} <111> slip systems. The ratio of yield stresses of (111) specimens to (100) specimens decreases rapidly from 1000 to 1600°C. Strain hardening for (111) specimens was much higher than for 100 specimens: The Burgers vectors of the active slip systems lie at 60 deg (or 120 deg) to each other and thus there are long range stresses between intersecting dislocations.
I. INTRODUCTION

The plastic deformation of crystalline solids is a subject of interest to the solid state physicist and to the materials scientist who desires to control the mechanical properties of engineering materials. In the last thirty years, much progress has been made in understanding plastic deformation in terms of the behavior of linear defects in the crystal lattice called dislocations.

Prior to 1948, experimental investigation of plastic deformation was limited to (a) macroscopic plastic strain rate measurements under various conditions of stress, strain, and temperature (the plastic strain rate of a specimen is proportional to the product of the length of dislocation moving in it and the average dislocation velocity), and (b) the study of surface features on deformed crystals such as slip steps, kinks, twins, deformation bands, etc., using the optical microscope. At that time it was not experimentally possible to obtain detailed information about dislocation behavior. In recent years, however, such detailed information has been obtained, through the development and application of several experimental techniques that make possible the direct observation of individual dislocations and their distribution in crystals during various stages of plastic deformation.

(a) One of these techniques is chemical etching. If a crystal containing dislocations is placed in a suitable etchant, etch pits are formed which correspond to the sites where dislocations meet the crystal surface. An etched crystal surface gives, therefore, a two-dimensional picture of the distribution of dislocations in the crystal. Although sometimes applied to metals, the etch pit technique has been most successfully used to study the distribution of dislocations in nonmetallic crystals such as LiF, MgO, and Si, because these materials can be grown with a relatively high degree of perfection.

(b) Another technique is that of thin-foil transmission electron microscopy. Originally applied to the study of dislocations in metals, this technique has recently been applied to several nonmetallic compounds as well. Contrast at dislocations is observed because of the strain field
of the dislocation, which locally reduces the electron transmittance of the foil. Magnification by the electron microscope is sufficient to resolve individual dislocation lines.

(c) A third technique for observing dislocations is to form an opaque precipitate on them. Dislocations treated in this manner are said to be "decorated" and can be seen with the optical microscope. This technique can, of course, be applied only to transparent crystals having a high degree of perfection.

(d) Finally, polarized light has been used to observe the distribution of dislocations in transparent, optically isotropic (cubic) crystals. If such a crystal is placed between crossed polarizers and properly oriented, the region around a slip band containing edge dislocations of predominantly one sign will appear light against a dark background. Light is transmitted through these regions because they are tetragonally distorted and therefore doubly refracting.

MgO, the material studied in this investigation, is optically isotropic and also transparent to the visible range of light. Single crystals of MgO are available commercially that have a high degree of perfection. Although a poor thermal conductor, MgO is sufficiently refractory to withstand electron beam heating in an electron microscope without significant deterioration. Thus, chemical etching, thin foil electron microscope, decoration, and polarized light techniques can all be used to study dislocation behavior in this material. Because of this wide variety of techniques applicable to it, MgO is an ideal material for investigating the relationship of dislocation behavior to the plastic deformation of crystalline solids.

Since 1958, the plastic deformation of single-crystal MgO has been extensively studied at room temperature. From this work a detailed picture of dislocation behavior during plastic deformation has been developed. Stokes, Johnston, and Li have shown that dislocation movement is initiated on \{110\} \{\overline{1}1\overline{0}\} slip systems by the expansion of dislocation half loops which are present at cleavage steps, scratches, and other points of damage at the crystal surface. Johnston and Gilman have measured the expansion velocities of individual dislocation
half loops in LiF and have shown that they vary with temperature and impurity content and are extremely sensitive to stress. Johnston has observed a similar behavior in MgO.

As a dislocation loop expands, it leaves in its wake "debris" in the form of elongated edge dislocation pairs or dipoles, which have been directly observed by Washburn and co-workers in thin foils of MgO with the electron microscope. These dipoles obstruct further dislocation movement on active slip planes but also provide the mechanism for spreading of mobile dislocation lines to adjacent atomic planes. Because dislocation movement within slip bands is obstructed by the presence of dipoles, most movement occurs at slip-band edges through undeformed regions. Hoover and Washburn have shown that, subsequent to yielding, the length of mobile dislocation in LiF is proportional to the number of interfaces between slip bands and undeformed material. Strain hardening was not observed until slip-band overlap and intersection became important.

Johnston and Gilman have developed a simple analysis that predicts the plastic yield behavior of LiF single crystals during a constant crosshead displacement rate test. In this analysis the slope of the stress-strain curve of a specimen is expressed as a function of its plastic strain rate. The plastic strain rate is predicted on the basis of the dislocation velocity and the length of dislocation line moving in the crystal. Recently, Johnston has used this analysis to predict the effect of varying such parameters as crosshead displacement rate, machine hardness, and dislocation velocity on yield behavior.

At elevated temperatures, much less is known about the relationship of dislocation behavior to the plastic deformation of MgO. Hulse and Pask, Thompson and Roberts, and May and Kronberg have studied the stress-strain behavior of MgO single crystals up to 1200°C.

The stress-strain data of these investigators show that the yield stress of MgO single crystals decreases with increasing temperature. Both the results of Hulse and Pask and of May and Kronberg show that annealing at 1000°C or above and then air quenching can reduce the yield stress by one-third to one-half. Of particular interest is a hump
at 600 to 700°C on the yield-stress-vs-temperature curve obtained by May and Kronberg with their annealed and quenched specimens. They attribute this hump to a precipitation hardening effect. No similar hump was observed by Hulse and Pask, probably because of differences in time that specimens were held at temperature prior to testing.

Hulse and Pask investigated the effect of stress rate on the stress-strain curve at room temperature. They found that the yield stress and the slope of the stress-strain curve subsequent to yielding could be increased slightly by increasing the stress rate. Similar behavior was observed by May and Kronberg by varying the crosshead displacement rate. They pointed out that a strain-rate-insensitive yield stress was consistent with the hypothesis that the dislocation velocity is very stress-sensitive.

All specimens tested by May and Kronberg exhibited a yield point followed by jerky flow at 900°C. Annealed and quenched specimens behaved in a similar manner at 600°C. Recently, Elkington, Washburn, and Thomas have observed similar behavior in four-point bend tests. They found that surface sources introduced at room temperature did not initiate slip-band formation above 500°C. Both May and Kronberg and Elkington et al. thus attributed this yield-point behavior to impurity pinning of dislocations.

The stress-strain curves of Hulse and Pask show an increase in slope subsequent to yielding with increasing temperature. The total strain at fracture of their specimens increased slowly from 26°C to 1000°C and rapidly thereafter.

Hulse, Copley, and Pask have obtained stress-strain curves up to 1250°C for single crystals of MgO stressed in a ⟨111⟩ crystallographic direction. Such specimens have no resolved shear stress on {110} planes. Above 350°C dislocation movement occurred on {100} planes. The resolved shear stress required for yielding on {100} planes was found to be 30 times that for {110} planes at 350°C. With increasing temperature, this stress decreased so that at 1200°C the ratio became about 3.5:1. The slopes of stress-strain curves of ⟨111⟩ oriented single
crystals, subsequent to yielding, were found to be many times as great as those with \( \langle 100 \rangle \) stress axes. The Burgers vectors of the active slip systems in this orientation lie at 60 deg to each other, and thus long-range forces exist between dislocations at slip-band intersections.

Information about the distribution of dislocations in MgO single crystals deformed at elevated temperatures is limited to the results of Elkington et al., who investigated the dislocation distribution in thin foils of MgO, taken from specimens deformed at -196 to 1200°C, using the transmission electron microscope technique. These investigators found, as was reported in Washburn's earlier papers, that the dominant feature of slip bands is the presence of many dislocation dipoles. These dipoles were found to decrease in number with increasing temperature. This decrease is consistent with the decrease in etch pit density observed in slip bands of LiF with increasing temperature by Johnston and Gilman.\(^2\) Most of the dipoles observed by Elkington et al. were nucleated at grown-in dislocations.

No measurements of the effect of temperature on dislocation velocities in MgO have been reported. Johnston and Gilman have made such measurements in LiF over the temperature range -196 to 26°C.\(^2\) They found that, for a given stress, the dislocation velocity increases with increasing temperature.

The purpose of this investigation has been to further elucidate the nature of plastic deformation of MgO single crystals at elevated temperatures. Carefully controlled stress-strain measurements have been obtained in compression at a constant stress rate at temperatures ranging from 1000 to 1600°C. These measurements have been correlated with studies of the distribution of dislocations in single crystals during various stages of deformation, which were determined with etch pit and stress birefringence techniques. Similar correlations have also been made in the temperature range 26 to 1000°C, where the stress-strain behavior of MgO has already been fairly well established by previous investigations. This brief review of the literature related to the plastic deformation of MgO and outline of the approach used in this investigation will be followed by a section describing experimental
procedures used in this investigation. This section will be followed by a results and discussion section divided into three parts. The first part deals with the problem of obtaining meaningful stress-strain measurements. The second part describes and discusses the distribution of dislocations in MgO single crystals deformed at elevated temperatures. In the third part, the results of stress-strain experiments are correlated with the observations described in the second part.
II. EXPERIMENTAL PROCEDURE

A. Apparatus

The initial stages of this investigation were concerned with designing and building an apparatus capable of precise stress-strain measurements at temperatures up to 1600°C at a constant stress rate. Compression loading was chosen over bending and tension for two reasons: (a) The stress distribution in an elastically compressed rectangular parallelepiped is both uniaxial and uniform; (b) no special specimen grips are required for the compression test, which would become a design problem for tensile testing at elevated temperatures. It was decided to test specimens in air so that direct measurement of strain in a central gage section of the specimen would be possible. This approach placed an upper limit on experimental test temperatures of about 1600°C, because of the unavailability of heating-element materials that would withstand a greater temperature in air. Constant stress-rate load application, and measurement of strain in a central gage section, were decided upon for reasons that will be discussed in Section III.A.

Figure 1 shows an overall view of the apparatus used in this investigation. Force is applied by a diaphragm cylinder A to a cylindrical column B, which is aligned by ball bearing bushings C. This column extends into the furnace D and transmits force to the specimen. Another load column enters the top of the furnace and is fastened to a triangular plate E, whose alignment will be described shortly. Strain is measured through a port in the side of the furnace by an electro-mechanical strain-measuring device F. Both strain and stress are measured with linear variable differential transformers (LVDT). The LVDT's are excited by Daytronic Differential Transformer units, G, [Daytronic Corporation, Dayton (Kettering) 29, Ohio]. These units also amplify the output signals of the LVDT's. These amplified signals are then fed into the channels of an X-Y recorder H. Thus, a continuous record of stress and strain during a stress-strain experiment is obtained. The temperature of the furnace is controlled to about ±5° by a current-adjusting three-mode proportional controller mounted in the rack J.
Fig. 1. Overall view of stress-strain apparatus.
The control current from this unit is amplified by a magnetic amplifier. This amplified signal is then used to control a 6-kVA saturable core reactor connected through a step-down transformer to the heating elements. The temperature of the furnace is measured by a single Pt/Pt-10%Rh thermocouple pressed against the side of the test specimen.

Figure 2 shows the furnace with one half rolled back to reveal the firing chamber. The test specimen (A) can be seen at the center of the furnace. Force is transmitted to it by the lower ram. Two alumina buttons separate the specimen from the loading rams (B) and act as stress distributors. The loading rams themselves are made from high-density 99.5% alumina and are fastened to the steel cylinders with water-cooled stainless steel connectors (C). Hairpin-shaped heating elements, made of MoSi2 and capable of withstanding temperatures up to 1700°C, can be seen hanging near the wall of the firing chamber. A protective coating of SiO2 forms on these elements when they are used in air. To the right of the specimen and pressing up against its surface is the thermocouple. To the left of the specimen and seated in two divot holes on its surface are pointed sapphire rods. These rods are lightly spring-loaded and connect to the pivot arms of the strain-measuring device. It can be seen that as the specimen deforms, the sapphire rods move together, thus causing the micrometer-mounted core (D) to move relative to the transformer coils (E). The translation of the core changes the coupling of opposing secondary coils with the primary coil, thus producing a voltage output proportional to the specimen strain. The alignment of the upper loading column by the triangular plate is clearly shown in this figure. The plate is held by opposing bolts. Those pressing against the plate have feet coupled to them by ball and socket joints.

Figure 3 shows the loading mechanism in greater detail than in Fig. 1. The control valve (A) is capable of supplying 0 to 100 psi air pressure to the pressure tank (B), which is almost filled with oil. During a stress-strain test, pressure is transmitted by an oil line from the pressure tank to the diaphragm cylinder (C). The diaphragm cylinder has an effective end area of 50 in.², and thus 100 psi can
Fig. 2. View of stress-strain apparatus with furnace opened.
Fig. 3. Loading mechanism for stress-strain apparatus.
produce a force on the loading column of 5000 lb.

During a stress-strain test, the stress on the specimen is obtained indirectly by measuring the pressure in the diaphragm cylinder with a Bourdon tube pressure transducer (D), which consists of an LVDT mounted on the end of a Bourdon tube. The stress system is initially calibrated by (a) making a load cell with resistance strain gages, (b) calibrating the output of this cell with a dead weight machine, and (c) using this cell to obtain a plot of force on the load column vs output of the pressure transducer. A linear relationship is obtained with no hysteresis. Thus, by suitably adjusting the amplification in the force channel, it is possible to read force directly on the X-Y recorder.

Various constant force rates are effected by coupling a constant-speed motor (E) to an adjustable-speed transmission (F). The transmission drives a micrometer (G) which depresses the actuator on the pressure valve at a constant rate. The response of the pressure valve is linear, and thus a constant loading rate is obtained. The response time of the system is quite rapid, depending on the setting of the bleed valve (H) on the delivery side of the pressure-regulating valve.

A relay valve is placed on the line connecting the pressure chamber to the diaphragm cylinder. This valve is operated by a switch on the loading column so that if the specimen breaks during loading, displacement of the loading column causes the relay valve to close, thus stopping the loading column.

B. Experimental Methods

1. MgO Single-Crystal Specimens

Specimens, with typical dimensions of 0.225 by 0.225 by 1 in., were prepared from aggregates of large MgO crystals obtained from the Norton Co., Worcester, Massachusetts, and from the Muscle Shoals Electrochemical Corp., Tuscumbia, Alabama. Spectroscopic analyses indicated for Norton Co. crystal—Fe, 0.04; Mn, 0.0015; Al, 0.005; Cu, 0.0015; Ca, 0.01; and Cr, 0.001%; and for Muscle Shoals Electrochemical Corp. crystal—Si, 0.004; Fe, 0.006; Mn < 0.0008; Al, 0.005; Cu, 0.001; Ca, 0.12; and Cr, 0.002% (values are reported
as oxides of indicated elements).] The (100) loading axis specimens were formed by cleaving; the (111) loading axis specimens were cut as described previously. After final shaping by grinding with 220A sandpaper, all specimens were polished for 1 min in 85% orthophosphoric acid at 110°C to remove part of the damage. Stress-strain curves obtained under the same experimental conditions for crystals from both sources were found to be quite similar.

2. Stress-Strain Tests

All specimens were loaded in compression and at a constant stress rate of 20 psi/sec unless otherwise stated. The reported stresses were calculated from initial specimen cross sections and are actual compressive stresses. Strains were determined by measuring the displacement of two small divot holes initially 0.5 in. apart centered on a side face of the specimens. All reported strains are true strains, the true strain being equal to the natural logarithm of one plus the engineering strain.

Specimen ends were constrained from translation in a plane perpendicular to the loading axis. This was effected by anchoring the alumina buttons, which acted as stress distributors, to the loading rams. A strong bond was found to develop at the specimen-button interfaces in spite of thin sheets of platinum placed there as reaction barriers. Thus, constraining the buttons constrained the specimen ends as well.

3. Chemical Etching

The etch used throughout this investigation consists of two parts 85% orthophosphoric acid and one part of concentrated sulfuric acid. (The writer is grateful to Jack Mitchell for suggesting this etch.)

Etching was carried out at room temperature. Etching for short periods of time (1 to 3 min) revealed only the "fresh" dislocations, i.e., dislocations resulting from damage to the surface; for longer periods, grown-in dislocations were revealed as well.
4. Stress Birefringence

When an unstressed optically isotropic crystal such as MgO is viewed through crossed polarizers, no light is transmitted through the system. If there is a stressed region in the crystal, however, light passing through this region becomes plane polarized along the principal stress axes. Upon emerging from the crystal the polarized waves have a phase difference $\delta$ given by

$$\delta = \frac{2\pi}{\lambda} c (\sigma_1 - \sigma_2) d,$$

where $\lambda$ is the wavelength of the incident light, $c$ is the stress-optical constant of the crystal, $\sigma_1$ and $\sigma_2$ are the principal stresses, and $d$ is the thickness of the crystal normal to the light path. Thus, the emerging wave front will, in general, be elliptically polarized and have a component along the analyzer axis.

If the polarizer axis makes an angle $\theta$ with one of the principal axes, the intensity of transmitted light, $T$, may be shown to equal

$$T = A^2 \sin^2 (2\theta) \sin^2 (\delta/2),$$

where $A$ is the amplitude of the incident light. It can be seen that, for a given phase difference, maximum birefringence is obtained by orienting the polarizer at 45 deg to one of the principal axes.

Slip bands, containing edge dislocations of predominantly one sign, have principal axes lying parallel and perpendicular to the Burgers vector. This condition follows from the fact that the shear stress at the midplane between two dislocations of the same sign and lying on the same plane is zero. Thus, in MgO, the principal axes for this configuration of dislocations lie along $\langle 110 \rangle$ directions. Also, the stress field at a slip band edge in MgO due to intersection by dislocations moving on the 90 -deg plane will have principal axes lying in the $\langle 110 \rangle$ directions. On the basis of these considerations, all stress birefringence photographs were taken through crossed polarizers with the polarizer axis lying along a $\langle 100 \rangle$ direction.
5. Photomicrography

All etch pit photographs were taken on a Leitz Metallographic Microscope (MM5) using 4 × 5-in. Polaroid type 55P/N film. The birefringence photographs were taken with a Praktica 35-mm camera through extension tubes and Kodak Plus X film.
III. RESULTS AND DISCUSSION

A. The Uniaxial Compression Test

The purpose of a stress-strain experiment is to measure the plastic strain rate of a volume of material under a variety of experimental conditions such as stress, strain, and temperature. In theory, such a strain rate can be directly related to dislocation behavior. Under actual experimental conditions, however, the interpretation of stress-strain curves in terms of strain rate and dislocation behavior is not straightforward. Complications arise because plastic deformation is an inhomogeneous process. The resistance to dislocation motion varies from region to region in a test specimen owing to differences in dislocation density. Also, in tension or compression tests a complex stress distribution exists near the ends of a test specimen. Thus, it is difficult to obtain strain rate measurements under well-defined and uniform experimental conditions.

In a compression experiment, additional complications arise because the ends of a test specimen often tend to translate relative to each other over the parallel surfaces of the loading rams. Such translation decreases the amount of dislocation movement on nonparallel slip planes and thus decreases the number of dislocation intersections. It can occur in spite of strong friction forces which oppose it, and can be avoided only by using some positive form of end constraint.

In addition, such a single crystal with its ends kept parallel and constrained from translation in a plane perpendicular to the loading axis can compress only by shear deformation if certain conditions are satisfied. These conditions depend on the assumed distribution of shear in the test specimen and are discussed in detail in Section III. A. 3; determination of the plastic strain rate from the stress-strain curve, however, is discussed first.
1. A Comparison of the Constant-Force-Rate and the Constant-Displacement-Rate Methods of Loading

In order to discuss the determination of the plastic strain rate from the stress-strain curve, it is necessary to distinguish between the constant-force-rate and the constant-crosshead-displacement-rate (CCDR) methods of loading. Although stress-strain data obtained by using both methods of loading have been reported in the literature, a comparison of these methods has not yet been made.

a. Constant force rate

The constant-force rate experiment is one in which the specimen is loaded by increasing the force acting on it at a constant rate. The difference between the true applied stress and that calculated by dividing the applied force by the undeformed cross section is small for the first few percent strain. In this range, constant force rate loading may be safely regarded as constant stress rate (CSR) loading.

The slope of the stress-strain curve obtained by constant force rate loading is then related to the plastic strain rate for the first few percent strain by the equation

\[
\frac{d\sigma}{d\varepsilon_p} = \left(\frac{d\sigma}{dt}\right)\left(\frac{d\varepsilon_p}{dt}\right)^{-1},
\]

where \(\sigma\) is the applied stress, \(\varepsilon_p\) is the plastic strain of the specimen, and \(t\) is time. It can be seen that a specimen loaded at a constant stress rate will always exhibit a positive slope unless its plastic strain rate is infinite. It can also be seen that linear strain hardening occurs only if the plastic strain rate of the specimen remains constant as the stress increases at a constant rate. Figure 4 shows a stress-strain curve with a shape typical of that observed in constant stress rate experiments on MgO single crystals at room temperature. The variation of strain rate with stress is also schematically shown, and is in accordance with Eq. (3).
Fig. 4. The variation of strain rate during a stress-strain experiment at constant stress rate.
b. Constant displacement rate

In the constant displacement rate test, the specimen is loaded by a crosshead which is displaced at a constant rate by some type of mechanical drive. The stress is usually recorded as a function of crosshead displacement. The nominal strain calculated by dividing the crosshead displacement by the undeformed length of the specimen is not equal to the plastic strain of the specimen, because the crosshead displacement is taken up by both elastic and plastic straining of the specimen and by the elastic flexing of the machine's crosshead. The latter contribution is by no means small. The force constant for crosshead flexing on conventional machines is about $5 \times 10^4$ lb. Thus, a force of 100 pounds, which corresponds to a stress of 1000 psi on a specimen with a cross-sectional area of 0.1 in. $^2$, would produce an elastic flexing of 0.002 in.

If the effect of crosshead flexing is properly taken into account, an equation relating the slope of a stress-strain curve in a constant crosshead displacement rate experiment to the strain rate of the specimen can be derived. For stress-strain curves in which the stress is plotted vs the nominal strain, this equation is

\[
\frac{d\sigma}{d\varepsilon_N} = \frac{L_0 K}{A} \left\{ 1 - \frac{d\varepsilon_p}{dt} \left( \frac{d\varepsilon_N}{dt} \right)^{-1} \right\},
\]

where $L_0$ is the initial specimen length, $A$ is the initial specimen cross section, $K$ is the force constant of the testing machine, $d\sigma/d\varepsilon_N$ is the slope of the stress-strain curve, $d\varepsilon_N/dt$ is the nominal strain rate, and $d\varepsilon_p/dt$ is the plastic strain rate. For stress-strain curves in which the stress is plotted vs the plastic strain, the proper equation is

\[
\frac{d\sigma}{d\varepsilon_p} = \frac{L_0 K}{A} \left\{ \left( \frac{d\varepsilon_N}{dt} \right) \left( \frac{d\varepsilon_p}{dt} \right)^{-1} \right\}. \tag{5}
\]
The plastic strain as plotted in such a curve can always be obtained, provided that $K$ is known, by subtracting the contribution of the elastic flexing of the crosshead from the nominal strain. The stress rate during a CCDR experiment is given by the equation

$$\frac{d\sigma}{dt} = \frac{L_0 K}{A} \left\{ \frac{d\varepsilon_N}{dt} - \frac{d\varepsilon_p}{dt} \right\}. \tag{6}$$

Equation (4) was previously derived by Johnston and Gilman but was not presented in the simple form given above. The derivations of Eqs. (4), (5), and (6) are given in Appendix A.

Figure 5 shows a stress-strain curve with a shape typical of that observed in CCDR experiments of LiF single crystals at room temperature. The variation of plastic strain rate and stress rate with nominal strain are also schematically shown, and are in accordance with Eqs. (4) and (6), respectively.

It can be seen that in the elastic region of the stress-strain curve, where the plastic strain rate is 0, the slope of the stress-strain curve is equal to $L_0 K/A$. As plastic deformation begins, the plastic strain rate increases rapidly, thus causing the stress rate to start to decrease from its initial value of $(L_0 K/A)\dot{\varepsilon}_N$. The stress rate decreases because the crosshead displacement is no longer taken up entirely by crosshead flexing. Some is now being taken up by plastic straining of the test specimen. The plastic strain rate continues to increase until it becomes equal to the nominal strain rate. At this point, the slope of the stress-strain curve is zero, as is the stress rate, because all the crosshead displacement is being taken up by plastic straining of the specimen. If the plastic strain rate continues to increase, a yield drop will occur; the stress rate becomes negative because the specimen is now taking up elastic strain stored in the flexed crosshead in addition to all the crosshead displacement. The decreasing stress eventually causes the strain rate to pass through a maximum and to start to decrease, which occurs at the inflection point on the high-strain side of the yield hump. At this point, the stress rate passes
Fig. 5. The variation of stress rate and plastic strain rate during a stress-strain experiment at constant displacement rate.
through a minimum. The yield drop continues until the plastic strain rate again equals the apparent strain rate, causing the slope of the stress-strain curve to be zero. Linear hardening occurs if the strain rate remains constant as the stress increases at a constant rate.

c. Summary

From this discussion, it can be seen that the CCDR and the CSR experiments are closely related. Both types measure the plastic strain rate of a test specimen under various conditions of stress and strain. The relationship between the slope of the stress-strain curve and the plastic strain rate is most straightforward, however, in the CSR experiment.

In the limit of an infinitely soft machine \((K \rightarrow 0)\), the CCDR experiment becomes a CSR experiment. In the limit of an infinitely hard machine \((K \rightarrow \infty)\), a CCDR experiment is a CSR experiment only when the slope of the stress-strain curve is constant. A slowly changing slope in this case corresponds to a rapidly varying stress rate. In machines of moderate hardness \((K \approx 5 \times 10^4 \text{ psi})\) (a category which includes most testing machines with crossheads), the CCDR experiment closely approximates a CSR experiment in regions where the slope of the stress-strain curve changes slowly. Thus, at very small plastic strains a specimen 1 in. long and 0.1 in. \(^2\) in cross section will behave the same when loaded in a CCDR test at a nominal strain rate of 0.5%/min as when loaded at a constant stress rate of approximately 40 psi/sec, as calculated according to

\[
\dot{\varepsilon} = \frac{L_0 K}{A} \varepsilon_N = \frac{1 \times 5 \times 10^4 \times 5 \times 10^{-3}}{10^{-1} \times 60} \approx 40 \text{ psi/sec}.
\]

2. Plastic Deformation under Inhomogeneous Conditions

In compression experiments, a problem arises because prior to plastic deformation the stress distribution in the ends of the specimen is usually different from that in its central region. Such a difference occurs either because the specimen ends are not flat or because they
are not initially parallel to the faces of the loading rams. In the latter case, an elastic bending of the entire specimen may occur. Because of this nonuniformity in the initial stress distribution, slip bands usually form first in the ends of a compression specimen.

Figure 6a shows an MgO single crystal that was strained slightly in compression at room temperature and then unloaded and photographed between crossed polaroids with transmitted light. It can be seen that slip bands have formed only in the ends of the specimen. Figure 6b shows another specimen, which was strained slightly more; in this case strain has occurred in the central part of the specimen as well.

Figure 7a shows two stress-strain curves obtained by compressing a single crystal of MgO, 1 in. high, at room temperature at a constant stress rate. In curve A, the stress is plotted vs the overall strain of the specimen. In curve B, the stress is plotted vs the strain in a central, 0.5-in. gage section. Curve A shows a lower yield stress than curve B, indicating that slip had occurred first in the ends of the specimen. The ends, however, hardened rapidly, so that once bulk yielding occurred, straining took place mostly in the central region.

The stress axis in a CSR experiment plot is also a time axis. Thus, at any instant of time the strain in the ends (0.25-in. sections) can be calculated from the stress-strain curves in Fig. 7a and compared with that in the central 0.5-in. gage section. Such a comparison is shown in Fig. 7b. The mismatch indicates very clearly that an inhomogeneous distribution of shear strain existed at all stages of deformation.

Figure 8 shows the stress-strain curves for another pair of single-crystal specimens of MgO cleaved from the same crystal block and stressed in a (100) direction. Curve A is for a crystal whose dimensions were 0.25 × 0.25 × 0.75 in.; the strain is the overall strain of the specimen. Curve B is for a crystal whose dimensions were 0.25 × 0.25 × 1.00 in.; the strain is the strain measured in a central 0.5-in. gage section. These curves indicate again the diversity of behavior that can be observed by using different measuring techniques, because the shear strain in compression specimens is not uniformly distributed.
Fig. 6. Stress birefringence of specimens deformed at room temperature. a. Loaded to a stress just less than the yield stress. b. Strained 0.1%.
Fig. 7. a. Stress-strain curves obtained by measuring overall and gage strain simultaneously.

b. A comparison of the strain in the ends vs the strain in the central gage section calculated from Fig. 7a.
Fig. 8. The effect of specimen height on the shape of the stress-strain curve.
The nonuniform distribution of shear strain that develops when a single-crystal specimen is compressed greatly complicates the interpretation of CCDR experiments obtained in compression. Strain rates obtained in this type of test do not correspond to a well-defined set of experimental conditions, because it is the overall strain that is normally measured. If strain is measured, instead, in a central gage section (in the same way as curve B in Fig. 8 was measured), a CCDR experiment does give strain rates under well-defined conditions. In this case, however, another complication arises, because at any instant of time the stress applied to the volume in which strain is measured is determined by the overall specimen strain and not by the strain observed in the gage section itself.

On the basis of the considerations described above, the CSR test with strain measured in a central gage section would appear to be the most direct method for making strain rate measurements in compression. Examination of Fig. 6 suggests that the central gage section should be at least far enough away from the specimen ends so that no slip planes lying within its bounds intersect the ends of the specimen. The sizes, shapes, and gage section placement for all specimens whose stress-strain curves were obtained in this investigation satisfied this condition.

3. The Effect of End Constraint

As was mentioned in Section III.A, the ends of a compression specimen often translate across the parallel surfaces of the loading rams during plastic deformation unless some kind of positive end constraint is applied. The application of such constraint, however, introduces a complication, because a single crystal with its ends kept parallel and constrained from translation cannot, in general, deform along an arbitrary stress axis. The conditions that must be satisfied for such deformation to occur depend on the distribution of shear in the specimen; two shear distributions will be discussed as limiting cases. First: The crystal is in a state of uniform strain due to continuous shearing on all slip planes of each slip system throughout its
volume. Second: The crystal is not uniformly strained. Continuous shearing occurs on all slip planes of each slip system except those intersecting the base of the crystal.

a. Uniform strain

The displacement \( u_i \) of an arbitrary point \( x_i \) due to a continuous shear \( dy^a \) parallel to a plane whose normal is \( \{ n_i^a \} \) and in a direction \( \{ t_i^a \} \) is given by

\[
u_i^a = \frac{3}{j=1} dy^a n_j^a x_j t_i^a ,\]

where the index \( a \) is used to label slip systems. The total displacement of a point due to shear on all active slip systems is

\[
u_i = \frac{n}{a=1} u_i^a .\]

If the total displacement components are known, the strains can be obtained by differentiation,

\[
\epsilon_{ij} = \frac{1}{2} \left\{ \frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i} \right\} .
\]

This analysis is valid only for infinitesimally small strains; however, it can be applied to large strains by viewing the components of the strain tensor as strain rates multiplied by an infinitesimal length of time.

Once the strains have been obtained from the shears, relative to some orthogonal set of axes, it is possible to determine whether or not the crystal can compress along one of these axes with its ends kept parallel and constrained from translation. The specimen can deform in this manner along, for example, the 3-axis only if it is possible to generate the strain components

\[
\epsilon_{33} = \text{compressive strain},
\]

\[
\epsilon_{12} = \epsilon_{13} = \epsilon_{23} = 0
\]

with the shears of the active slip systems.
As an example, the components of the strain tensor resulting from shearing MgO on the \{110\} \langle1\overline{1}0\rangle family of slip systems will be calculated, referenced to the cube axes, using Eqs. (7) to (9). They are as follows:

\[
\epsilon_{11} = \frac{\partial u_1}{\partial x_1} = -\frac{1}{2} \gamma^3 - \frac{1}{2} \gamma^4 + \frac{1}{2} \gamma^5 + \frac{1}{2} \gamma^6 ,
\]

\[
\epsilon_{22} = \frac{\partial u_2}{\partial x_2} = -\frac{1}{2} \gamma^1 - \frac{1}{2} \gamma^2 - \frac{1}{2} \gamma^5 - \frac{1}{2} \gamma^6 ,
\]

\[
\epsilon_{33} = \frac{\partial u_3}{\partial x_3} = \frac{1}{2} \gamma^1 + \frac{1}{2} \gamma^2 + \frac{1}{2} \gamma^3 + \frac{1}{2} \gamma^4 ,
\]

\[
\epsilon_{12} = \frac{1}{2} \left( \frac{\partial u_1}{\partial x_2} + \frac{\partial u_2}{\partial x_1} \right) = 0 ,
\]

\[
\epsilon_{13} = \frac{1}{2} \left( \frac{\partial u_1}{\partial x_3} + \frac{\partial u_3}{\partial x_1} \right) = 0 ,
\]

\[
\epsilon_{23} = \frac{1}{2} \left( \frac{\partial u_2}{\partial x_3} + \frac{\partial u_3}{\partial x_2} \right) = 0 .
\]

The shears are labeled 1 to 6 corresponding to the (011)[0\overline{1}1], (0\overline{1}1)[011], (101)[\overline{1}01], (\overline{1}01)[101], (1\overline{1}0)[1\overline{1}0], and (1\overline{1}0)[110] slip systems, respectively. One additional relationship exists between the components of strain, namely

\[
\epsilon_{11} + \epsilon_{22} + \epsilon_{33} = 0 ,
\]

which expresses the fact that there is no volume change during a shear deformation. Thus, only two components of the strain tensor can be independently adjusted by shear on the \{110\} \langle1\overline{1}0\rangle slip systems. 17

It can be seen by comparing (i) with (ii) that, by slipping on the \{110\} \langle1\overline{1}0\rangle slip systems, MgO can deform in compression along a cube axis with its ends parallel and constrained from translation. To
find out whether or not it could undergo a similar deformation along 
some other stress axis, it is necessary to refer the strain components 
(ii) to a new set of basis axes running along the edges of a compression 
specimen with the new stress axis (the specimen is assumed to be a 
rectangular parallelepiped). This can be done with the equation

$$\epsilon'_{im} = a'_{i} a_{mj} \epsilon_{ij},$$

where

$$a'_{i} = \hat{e}'_{i} \cdot \hat{e}_{i}$$

and \(\{\hat{e}'_{i}\}\) and \(\{\hat{e}_{i}\}\) are the new and old basis vectors, respectively.
The summation convention is to be applied in Eq. (10). If conditions 
(i) can be satisfied by the new strain components, then compressive 
deformation can occur, in the same manner as before. In general, 
these conditions can be satisfied for all orientations of stress axis only 
if the crystal has five independent slip systems. (A slip system is 
independent if its operation produces a change in shape that cannot be 
produced by slip on the other systems.)

b. Nonuniform strain

A specimen with no shear on slip planes intersecting its ends is 
a better model of how slip actually occurs in a compression specimen. 
In this case the ends must remain parallel no matter what the distribu­
tion of shear in the specimen. Specimens can compress along a stress 
axis \(PQ\) with the ends kept parallel and constrained from translation 
if two conditions are satisfied:

(a) The active slip systems must be able to generate a compressive 
strain along \(PQ\) as determined by

$$\epsilon_{PQ} = \epsilon_{j k} \nu_{j} \nu_{k},$$

where \(\{\nu_{j}\}\) are the direction cosines of the axis \(PQ\) and the summation 
convention is to be applied. The strain components \(\epsilon_{j k}\) are calculated 
by using Eqs. (i) to (iii).

(b) The possible translation vectors of an arbitrary point in a plane
perpendicular to the loading axis must be linearly dependent.

The actual distribution of shear in a deformed single crystal is best approximated by a mixture of the two limiting cases described here. All stress-strain tests carried out in this investigation were consistent with the condition for both types of shear distribution.

B. The Distribution of Dislocations in MgO Single Crystals Deformed at Various Temperatures

In Section III. A the problem of making meaningful strain rate measurements was considered. In Section III. C the results of such measurements and their interpretation will be given. Stress-strain data, however, provide information only about the total length of dislocation line moving in a crystal and its average velocity. In order to properly interpret such data, a knowledge of the distribution of dislocations during various stages of deformation is necessary. In this section the results of etch pit studies and stress birefringence studies of the dislocation distributions in single crystals of MgO deformed from 26 to 1600°C are reported and discussed.

1. The Relationship of Surface Sources to the Distribution of Dislocations at Room Temperature

Undeformed single crystals are never perfect, but contain a grown-in substructure formed during crystal growth. This substructure generally consists of networks of dislocations forming subgrain boundaries and also of randomly spaced individual dislocations. The subgrains of commercially available MgO thus formed are about 1 to 10 mm in diam. Within these subgrains dislocation densities of about $5 \times 10^4$ cm/cm$^3$ are observed. 12

For many years it was believed that plastic flow was initiated in undeformed crystals by the bowing out of segments of grown-in dislocation line. 18 Frank and Read had shown how such segments could act as generators of mobile dislocation loops. 19 Their mechanism provided an explanation for the appearance of slip steps on the surfaces of deformed metal crystals many thousands of times as large as could have been formed by a single dislocation.
It is now, however, generally accepted that, at room temperature, slip is not initiated by grown-in dislocations in ionic crystals. Gilman has shown in LiF that dislocation half loops are present on crystal surfaces that can expand when stressed and initiate slip bands. These half loops are present at cleavage tears or can be nucleated by even slight impacts. Similar behavior has been observed in MgO. Also, in MgO, grown-in dislocations have been proven extremely immobile at room temperature. Stokes has reported an experiment in which single crystals of MgO were chemically polished, until all surface half loops were removed, and then tested in tension. One such specimen supported a tensile stress of 140,000 psi without deforming plastically. Because it has been demonstrated that a single mobile dislocation line (a half loop) can initiate a slip band, it is evident that no mobile dislocation lines were present up to a resolved shear stress of 70,000 psi in Stokes' specimen.

The way in which a single-surface half loop develops into a slip band in ionic crystals is not completely understood at present. Both a multiplication of the length of mobile dislocation line and a spreading of mobile dislocation line to planes adjacent to the active slip planes must occur. Several mechanisms, based on the formation by moving dislocation lines of dislocation configurations similar to the Frank-Read source, have been proposed to explain this behavior.

A number of investigations have been concerned with elucidating the formation and structure of slip bands in MgO. Several investigations have studied the formation of cleavage cracks at slip band intersections. Little consideration has been given, however, to how the distribution of damage half loops on a crystal surface might affect plastic deformation. Figure 9 shows the cleaved surface of an MgO single crystal etched to reveal the dislocations. Prior to etching, the crystal was loaded briefly to a stress just less than that required to cause yielding but large enough to cause dislocations to move and multiply. Dislocations can be seen moving away from various types of damage commonly found on the cleaved surfaces of MgO single crystals.
Fig. 9. Slip band initiation at (a) a rosette, (b) a cleavage tear, and (c) a scratch.
Figure 9a shows a dislocation rosette. Rosettes are formed by impact, and consist of dislocation damage lying on all six \{110\} planes, which explains their peculiar shape. This picture also shows a number of small half loops which have begun to multiply into slip bands. The curved lines of smaller pits are subgrain boundaries. Figure 9b shows dislocations moving away from a cleavage tear running diagonally across the picture. Markings of the crisscross type also shown in this picture can be produced by rolling a small steel ball across the crystal surface. Figure 9c shows dislocations moving away from a scratch.

In each of the pictures shown in Fig. 9, the micron marker lies along a \langle 100 \rangle direction. Dislocations lying along lines at 45 deg to the marker are in edge orientation where they emerge from the crystal, while dislocations lying along lines parallel or perpendicular to the micron markers are in screw orientation. It is interesting that in all three pictures the edge dislocations appear to be considerably more active than the screw dislocations. This condition is consistent with Johnston and Gilman's observations that in LiF the velocity of edge dislocations at a given stress exceeds the velocity of screw dislocations by a factor of 50:1.

A large number of slip band initiation sites in a crystal leads to the formation of many narrow bands. On the other hand, if a crystal is chemically polished until most of its initiation sites are removed, a few wide bands will form. Washburn and Gorum have reported that the shear strain in slip bands of MgO at room temperature is uniformly distributed and equal to a saturation value of 10% independent of slip band width. Thus, at a given strain, the total slip band width in the narrow-band specimen will equal that in the wide-band specimen. Thus, the widening of bands with increasing strain should be more apparent in specimens with a few wide bands than in specimens with many narrow ones.

Figure 10 illustrates how a decrease in the number of moving dislocations can occur during the broadening of many unequally spaced slip bands, whereas it would not occur in a specimen with few slip bands. The bands with horizontal lines in specimens A and B represent the
Fig. 10. Mechanism for decreasing $p$ by overlap of unequally spaced slip bands.
slip bands initially present. The bands with vertical lines correspond to regions sheared during band broadening. It can be seen that before and after band broadening occurred, the strain in specimen A was equal to the strain in specimen B. During slip band broadening, however, the amount of slip band interface and thus the length of moving dislocation line remains constant for specimen A, but for specimen B it decreases by a factor of 1/3. Thus, the number and distribution of slip band initiation sites on the surface of a crystal can affect the length of dislocation line moving in a crystal, and consequently its stress-strain behavior. In Section III. C stress-strain curves are presented that further illustrate this effect.

2. The Effect of Temperature on Slip Band Structure

Increasing the temperature of deformation can affect the structure of slip bands in MgO in a number of ways. As the temperature is increased, the mobility of dislocations increases. At elevated temperatures slip bands form at lower stresses on \{110\} planes than at room temperature. Although the mobility of dislocations on \{100\} planes is so low at room temperature that slip on these planes is not observed to occur, the mobility increases very rapidly with increasing temperature. At 350°C, yielding on \{100\} planes requires thirteen times as much stress as required for yielding on \{110\} planes; at 1600°C only 2.9 times as much stress is required. Also as the temperature increases, diffusion-controlled processes begin to occur at measurable rates. Thus, the ease of cross slip and climb and also the mobility of jogs increases with increasing temperature. Finally, the distribution and number of sites where slip bands can be initiated may change in a crystal at elevated temperature. Gilman has shown that in LiF single crystals quenched rapidly from an elevated temperature, the subgrain boundaries can act as initiators of slip bands. Stokes, on the other hand, has found the opposite to be true in the case of MgO at room temperature. Evidence found in this investigation that grown-in dislocations do act as slip band initiators in MgO at elevated temperature is presented in Section III. B. 3.
Figure 11 shows etched slip bands formed at the surface of MgO single crystals deformed at various temperatures and with various previous heat treatments prior to testing. The bands running parallel and perpendicular to the micron markers are screw bands; those running at 45 deg to the markers are edge bands. (Slip bands in which the emerging dislocations are in screw or edge orientation are called screw or edge-slip bands, respectively.) The surfaces of all the crystals shown here were cleaved and then polished for 5 min in orthophosphoric acid at 150°C prior to loading. Experience has shown that such treatment greatly reduces the number of slip bands initiated on the crystal surface, so that those that are formed must widen rapidly with increasing strain.

It can be seen that increasing the temperature of deformation decreased the etch pit density, $p$, in both the edge and screw slip bands. Annealing and then quenching prior to testing decreased the etch pit density in the edge band but not in the screw band. In all specimens except the annealed and quenched specimen the etch pit density in the edge slip bands was considerably greater than in the screw bands. Also of interest is the gradient of damage evident in the edge band formed at 1000°C. Etch pit counts were made for the slip bands shown in Fig. 11 and are reported in Table I.

Two additional features of Fig. 11 deserve comments. The first is the reduction of etch pit density that can be seen to occur in the screw bands at one side or the other of an edge band intersection. This condition prevails because a screw band lies at 45 deg to the crystal surface and thus is screened from the surface at one side of its intersection with an edge band. The second feature is the size of etch pits observed in the various slip bands. Isolated pits in the slip bands at 20°C and 500°C are about 0.75 $\mu$ (7500 Å) in diameter. Etching time for these specimens was 1 min. Larger pits with an average diameter of about 1.5 $\mu$ were formed in the slip bands of the specimen deformed at 1000°C. This specimen was etched for 5 min. Also present in the slip bands of the 1000°C specimen are some large flat pits about 1 to 2 $\mu$ in diam and a number of very small pits about 0.35 $\mu$ in diam. The etch pit size is a function of the kind of etch used, the time of etching,
Fig. 11. Etched screw and edge slip bands: (a) Specimen slow-cooled to 20°C where it was deformed; (b) specimen annealed at 1000°C and "quenched" to 20°C where it was deformed; (c) specimen deformed at 500°C; and (d) specimen deformed at 1000°C.
Table I. Summary of etch pit density shear strain measurements.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>$\varepsilon_{xx}$</th>
<th>$\tau_{\text{max}}$ (psi)</th>
<th>$\varepsilon_{xy}$</th>
<th>$\rho$ cm$^{-2}$</th>
<th>Region</th>
</tr>
</thead>
<tbody>
<tr>
<td>26°C slow cooled</td>
<td>0.03</td>
<td>9000</td>
<td>0.0635</td>
<td>$1.7 \times 10^8$</td>
<td>edge band</td>
</tr>
<tr>
<td>26°C quenched</td>
<td>0.005</td>
<td>5300</td>
<td>0.055</td>
<td>$1.3 \times 10^8$</td>
<td>edge band</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>screw band</td>
</tr>
<tr>
<td>500°C</td>
<td>0.005</td>
<td>3400</td>
<td>0.06</td>
<td>$1.1 \times 10^8$</td>
<td>edge band</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>$7.6 \times 10^7$</td>
<td>screw band</td>
</tr>
<tr>
<td>1000°C</td>
<td>0.005</td>
<td>2000</td>
<td>$-$</td>
<td>$5.1 \times 10^7$</td>
<td>edge band</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>screw band</td>
</tr>
</tbody>
</table>

a. Longitudinal strain.
b. Shear strain in slip bands.
c. Estimate of shear strain based on single band measurements of 0.06, 0.05, and a value of 5.44% obtained by averaging over several bands.
d. Estimate of shear strain based on a single band measurement of 6.43% and a value of 5.85% obtained by averaging over several bands.
e. Etch pit count made in region of high density at edge of band.
the purity of the crystal, and the location of impurities relative to the
dislocation as well as of the type of defect that is being etched. It is
believed that the large flat pits in the 1000°C specimen may corre-
spond to sites of impurity precipitates, as was observed by Stokes. The
smaller pits observed in this specimen as well as the other specimens
in regions of low etch pit density may correspond to smaller types of
defects such as small prismatic loops or vacancy clusters.

As previously mentioned, Washburn and Gorum have measured
the amount and the distribution of shear strain in slip bands at room
temperature. This was done by measuring the displacement of an edge
band formed early during deformation by an intersecting edge band
formed at a later time. Similar measurements have been carried out
as a part of this investigation at room temperature in quenched and
unquenched specimens and at 500°C. The results are given in Table 1.
No measurements were possible at 1000°C because the edge bands were
too diffuse.

Figure 12, for example, shows intersecting edge bands in a
specimen deformed at 500°C, etched, deformed at 500°C again, and
then re-etched. The large etch pits show the width of the slip bands
following the first deformation. The small pits are the result of a
widening of the slip band in the upper left corner, which occurred during
the second deformation. The slip band at 90 deg to this band was not
active during the second deformation and thus has acted as a marker
revealing the shear displacement due to the broadening of the upper
left band. As was observed by Washburn and Gorum at room tempera-
ture, the shear displacement in this band is also uniformly distributed.
The total shear strain is 0.0643.

Figure 13 shows edge and screw bands on the same specimen that
were quite narrow following the first deformation but that broadened
appreciably during the second one. The principal item of interest in
this picture is that most of the etch pits in the edge bands remained
sharp-bottomed during the second deformation, while those in the screw
bands did not. This indicates that most of the dislocations associated
with pits in the edge bands were immobile and did not move during the
Fig. 12. Slip band in specimen deformed 0.1% at 500°C, etched, deformed 0.5% at 500°C, and then re-etched.
Fig. 13. Mobility of dislocations at 500°C in (a) edge bands and (b) screw bands. Same specimen as in Fig. 12.
second deformation, while most of the dislocations in the screw bands did move.

This observation and the observation of higher etch pit densities in edge bands than in screw bands (except in the quenched specimen deformed at room temperature) are consistent with the observations of dipoles in thin foils of MgO. It has been pointed out by Washburn et al. that dipoles can be formed whenever a moving screw dislocation develops a jog large enough to pin it at a point. According to Washburn, the dislocation moves forward by leaving two parallel segments of edge dislocation line, with opposite signs, trailing back to the jog. If the separation of the two edge dislocations normal to their slip planes, $h$, is less than $h^*$, where

$$h^* = \frac{Gb}{8\pi(1-\nu)t},$$

and where $G$ is the shear modulus, $b$ is the Burgers vector, $\nu$ is Poisson's ratio, and $t$ is the resolved shear stress, then the edge dislocations are blocked by each other and form a dipole. Such dipoles, because they trail behind dislocations of near screw orientation, would lie in approximately a $\langle 100 \rangle$ direction and thus would appear predominantly in edge bands, increasing the etch pit density in them. Also, because the dipoles are immobile, it would be predicted that most pits in edge bands would remain sharp-bottomed during stressing, which is the exact behavior observed in Fig. 13. If $h$, on the other hand, is greater than $h^*$, the two edge dislocations may cross over each other and act as a source of mobile dislocation line. If such a process occurs at the edge of a slip band, the slip band widens.

Two predictions can be made, on the basis of Eq. (13), which can be experimentally checked:

1. The maximum dipole spacing observed in slip bands should not exceed $h^*$.
2. The minimum spacing for active slip planes should not be less than $h^*$.

The first prediction was checked by Elkington et al., who studied dipole
spacings in thin foils of MgO, taken from specimens deformed at various temperatures. They observed that the maximum dipole spacing increased with increasing temperature as would be predicted by Eq. (13), because yield stress decreases; however, the dipole spacing was never observed to exceed the calculated $h^*$. The second prediction will be used shortly to interpret the results of the etch pit density and shear strain measurements made in this investigation.

First, however, it will be necessary to consider in greater detail the application of Eq. (13) to the prediction of dipole and slip band spacings. A complication arises because $\tau$ in this equation should not be set directly equal to the resolved shear stress as calculated from the applied load at some stage of deformation, for this neglects the interaction forces between the dislocation line and various hardening centers present in the crystal volume. Johnston has presented convincing evidence that such hardening centers are present in LiF and are associated with the presence of Mg as an impurity in the crystal lattice. The strength of the interaction between such centers and the dislocation line should depend on their size and distribution and also on the line tension of the dislocation. Because of the great similarity of the plastic behavior of MgO to LiF, it is likely that such hardening centers exist in MgO as well. Thus, it is proposed that Eq. (13) should be changed to

$$h^* = \frac{G(T)b}{8\pi(1-\nu)[\tau - \tau_{SB}(T)]} , \quad (14)$$

where $\tau$ equals the resolved shear stress as calculated from the applied load, $\tau_{SB}(T)$ is a static back stress introduced to account for short-range stress interactions between the dislocation line and hardening centers, and the shear modulus is written as $G(T)$ to show explicitly that it depends on temperature.

The following example, although hardly representative of the actual three-dimensional case, does give some idea of the meaning of the back stress term. Let us consider the potential energy of a two-dimensional lattice containing an edge dislocation but otherwise perfect.
This energy depends on the position of the dislocation and can be crudely represented by

\[ V = \frac{1}{2} V_0 + \frac{1}{2} V_0 \sin \left( \frac{2\pi}{b} x - \frac{\pi}{2} \right), \]

where the origin is taken at an equilibrium position of the dislocation and the zero of energy is taken to be the strain energy of the crystal when the dislocation is in an equilibrium position. \( V_0 \) therefore equals the strain energy of the lattice when the dislocation is in a midglide position. This equation is plotted in Fig. 14a. If a shear stress is applied to the slip plane, each equilibrium position of the dislocation must be considered to have a slightly different potential energy. The energy difference between adjacent equilibrium sites is \( \tau b^2 \). In this case the potential energy of the system is given by

\[ V = \frac{1}{2} V_0 + \frac{1}{2} V_0 \sin \left( \frac{2\pi}{b} x - \frac{\pi}{2} \right) - \tau b x. \]

This equation is shown in Fig. 14b, and can be differentiated to obtain the force curve shown in Fig. 14c,

\[ F = -\frac{dV}{dx} = -\frac{\pi V_0}{b} \cos \left( \frac{2\pi}{b} x - \frac{\pi}{2} \right) + \tau b. \]

The crosshatched areas, then, represent the amount of thermal energy that must be supplied for the dislocation to move forward to the next equilibrium position. The velocity of the dislocation is given by the expression

\[ v = b v e^{-\Delta H(T)/RT}, \]

where \( v \) is a frequency on the order of the Debye frequency and \( \Delta H(T) \) is the activation energy.

Hardening can be introduced into this model by adding another periodic term to the potential so that it is given by

\[ V = \frac{1}{2} V_0 + \frac{1}{2} V_0 \sin \left( \frac{2\pi}{b} x - \frac{\pi}{2} \right) + \frac{1}{2} H \sin \left( \frac{2\pi}{nb} x - \frac{\pi}{2} \right) - \tau b x. \]
Fig. 14. Strain-energy and force curves of a crystal containing a dislocation (a) unstressed, (b) stressed, (c) force curve for stressed crystal, and (d) force curve of stressed curve with hardening centers.
This equation can be differentiated to obtain the force

\[ F = -\frac{dV}{dx} = \frac{\pi V_0}{b} \cos \left( \frac{2\pi x}{b} - \frac{\pi}{2} \right) - \frac{\pi H}{nb} \cos \left( \frac{2\pi x}{b} - \frac{\pi}{2} \right) + \tau b \]  

and is plotted in Fig. 14d.

It is obvious, upon comparing Figs. 14c and 14d, that at a given stress the dislocation will move slower in the lattice with the hardening centers. Both wavelength and amplitude of the hardening potential can affect its velocity. If \( \tau^*_T(T, v) \) is taken to be the stress required to move a dislocation at a given velocity in a perfect lattice, then \( \tau_{DB}(T, v) \), the dynamic back stress, may be defined as that stress which must be added to \( \tau^*_T(T, v) \) to produce the same velocity in the lattice with hardening centers. The resolved shear stress, \( \tau \), as calculated from the applied load must therefore equal the sum \( \tau_{DB}(v, T) + \tau^*_T(v, T) \).

In a similar manner, if \( \tau^*_{ST}(T) \) is taken to be the stress required to move the two edge dislocations of a dipole past each other in a perfect lattice, then \( \tau_{SB}^*(T) \), the static back stress, may be defined as that stress which must be added to \( \tau^*_{ST}(T) \) in order to produce the same effect in a lattice with hardening centers.

The dynamic and static back stresses are, in general, not equal. The dynamic back stress is a function of the size and distribution of the hardening centers (which would correspond to the amplitude and wavelength of the hardening potential in our simple model), and the dislocation velocity; it may be thermally activated. The static back stress depends, however, primarily on the size and distribution of hardening centers in the crystal. If these remain constant, its only temperature dependence results from the temperature dependence of the shear modulus. For this reason, it can be concluded that if the size and distribution of hardening centers remain constant, a decrease in \( \tau \) results in a decrease in \( \tau - \tau_{SB}^* \). Since \( \tau \) decreases with increasing temperature, \( \tau - \tau_{SB}^* \) decreases with increasing temperature and thus, by Eq. (14), \( h^* \) must increase. Therefore the spacing between active slip planes should increase. In this investigation the measurements of shear strain at 26°C and 500°C indicate no corresponding decrease,
and thus it can be concluded that the number of dislocations crossing each active slip plane has increased.

The amount of damage that accumulates on a slip plane should decrease with increasing temperature, because \( \tau - \tau_{SB} \) is the effective stress driving dislocations across the slip plane. Because the amount of damage left on a slip plane should be proportional to the number of dislocations that have crossed it, it can be concluded that moving dislocations must produce less damage as the temperature increases. A decrease in the damage produced by a moving dislocation could occur between 26 and 500°C because of the increase in dislocation mobility on \( \{100\} \) planes. Jogs which at 26°C might become trapped on moving dislocations of near screw orientation may be sufficiently mobile at 500°C to escape onto dislocation segments of near edge orientation, where they can easily move without forming dipoles.

Both (a) the decrease in etch pit density in the edge bands of the 500°C specimen relative to the 26°C specimen observed in this investigation, and (b) the observation by Elkington et al. that both the number of dipoles and the total amount of damage in slip bands decreases with increasing temperature, are consistent with the predictions of the preceding paragraph. A decrease in etch pit density was also observed in this investigation in the edge bands of the specimen deformed at 1000°C. In this case, however, a complication arises because of the gradient of damage in the edge band. A possible explanation is that the slip band had widened in the direction of increasing etch pit density and that many dipoles in the part of the band which formed first had annealed out.

The marked decrease in etch pit density in the screw bands of the 500°C specimen (relative to the 26°C specimen) observed in this investigation suggests that at room temperature large jogs on dislocations close to edge orientation may be immobile enough to form dipoles at 26°C but not at 500°C. This would also be consistent with the fact that the screw bands in the quenched specimen at 26°C did not decrease in density relative to those of the unquenched, because in this case no increase in dislocation mobility on \( \{100\} \) planes occurred.
The decrease in etch pit density in the edge bands of the quenched specimen, although not predictable because a change of the size and distribution of the hardening centers was probably induced by quenching, is consistent with Eq. (14). Because $\tau^*(v, T)$ should be the same in the quenched and unquenched specimens, it can be concluded that $\tau_{DB}(v, T)$ is less in the quenched specimens than in the unquenched. Thus, it would appear that quenching decreases $\tau_{DB}(v, T)$ without decreasing $\tau_{SB}$ proportionately.

This discussion of the effect of temperature on slip band formation and structure has by necessity been somewhat speculative in nature. Much information such as the actual spacing of slip bands at various temperatures is still to be obtained. The purpose of this discussion has been to indicate how the observations made up to the present might eventually fit together into a self-consistent theory.

3. The Distribution of Dislocations in Single Crystals

Deformed at 1000°C

Although slip bands form in MgO deformed 0.5% at 1000°C, a new phenomenon was observed at this temperature which profoundly affects the distribution of dislocations in such crystals. Figure 15a shows the etched surface of an MgO single crystal deformed 0.5% at 1000°C. Several screw slip bands and edge slip bands, running respectively perpendicular and at 45 deg to the micron marker, are clearly visible. Slip of a more uniform variety, however, appears to have occurred throughout the volume of the crystal as well.

The most straightforward explanation for the distribution of slip observed in this crystal is that plastic deformation was first initiated at regions of surface damage so that slip bands were formed. Before these slip bands could grow very wide, however, plastic deformation was initiated at a large number of other sites throughout the volume of the crystal. On the basis of Gilman's quenching experiments on LiF (in which he showed that grain boundaries became mobile in this material after quenching), and because dislocations are quite mobile on \{100\} planes in MgO at this temperature, it is believed that the initiation
Fig. 15. a, b, and c. Etched single crystal strained 0.5% at 1000°C.
sites for this secondary slip are segments of the grown-in dislocation substructure.

It can be seen in Fig. 15a that the primary slip bands act as barriers to the secondary slip. In the regions of intersection between the primary bands and the secondary slip, the secondary slip dislocations tend to polygonize.

Figure 16 shows a specimen deformed 0.1% at 1000°C as seen through crossed polaroids. The birefringence distribution observed in this specimen differs considerably from that observed in the specimen deformed 0.1% at room temperature shown in Fig. 6b. In general, it exhibits less intense birefringence than the specimen deformed at room temperature, and the slip bands where they appear are less well defined. The decrease in birefringence is undoubtedly due to the fact that most of the dislocation damage is now spread uniformly throughout the crystal rather than being concentrated into slip bands. Also, regions of intersection between the primary bands and secondary slip may have a characteristic birefringence associated with them that contributes to the overall lack of sharpness.

In regions of intersection between secondary slip on slip planes at 90 deg and at 45 deg to the crystal surface, dislocation boundaries were observed to form in the same crystal shown in Fig. 15a. Figure 15b shows such a boundary. It can be seen that the dislocations forming this boundary tend to line up in a (100) direction. Such boundaries were not always observed to lie parallel to the trace of the active slip planes at 45 deg to the crystal surface. They were often observed to be perpendicular to this direction or to be curved, as shown in Fig. 15c. It is evident that this particular boundary formed after some deformation had taken place, because its growth appears to have been impeded by the slip band.

In Section III. B. 4, the nature of these boundaries is discussed in more detail. It is shown that at large strains and high temperatures they lead to the formation of deformation bands clearly visible on the surfaces of the deformed crystals.
Fig. 16. Specimen strained 0.1% at 1000°C as seen with transmitted light through crossed polarizers.
4. The Distribution of Dislocations in Heavily Deformed Crystals at Temperatures Greater than 1250°C

At temperatures in the neighborhood of half the melting point---about 1250°C in MgO---diffusion should start to play an important part in the deformation process. [Calculation of "half the melting point" is based on the absolute temperature scale; for example, if the melting point of MgO is taken as 3073°K (2800°C) half the melting point is 1536°K (1263°C).] Dislocation climb provides an additional degree of freedom by which dislocations of the same Burgers vector but of opposite sign can annihilate each other, and by which dislocations of the same Burgers vector and the same sign or dislocations with different Burgers vectors can rearrange themselves into configurations of lowest strain energy. Because of such annihilation and rearrangement, no useful information was obtained in this investigation by etching specimens that had strained only a small amount at 1300°C and above. It has, however, been possible to obtain a number of pictures illustrating the distribution of slip in heavily deformed specimens at temperatures of 1300°C and above, by use of the etch pit and stress birefringence techniques. Also, examination of deformation bands and kinks on the surfaces of deformed specimens and steps on internal surfaces produced by cleaving has provided additional useful information.

Figure 17a shows a typical etch pit configuration observed on the outer surfaces of specimens deformed about 15% at 1300°C. It can be seen that at this temperature and strain the etch pit density is quite high and the etch pits are uniformly distributed. The average density of etch pits on this surface was $3.3 \times 10^7$ pits/cm$^2$. The dark lines which can be seen running in \langle110\rangle directions across the surface of this specimen are discussed shortly.

Figure 17b shows a typical etch pit configuration observed on the outer surfaces of specimens deformed about 15% at 1600°C. The average etch pit density on this surface was $1.1 \times 10^7$ pits/cm$^2$. Specimens deformed at both 1300 and 1600°C were cleaved to provide internal surfaces for examination. These surfaces were then chemically polished and etched. The etch pits observed on these surfaces were
Fig. 17. Specimens strained 15% (a) at 1300°C, and (b) at 1600°C.
uniformly distributed. No ordering of etch pits was observed.

a. Interactions between dislocations on orthogonal slip systems

Figure 18 shows a higher magnification of a region near one of the dark lines in Fig. 17a. It can be seen that these lines correspond to closely spaced pits and thus mark the intersection of dislocation boundaries within the crystal with the crystal surface. It is also of interest that the etch pits at this temperature tend to be flatter. This is probably due to the segregation of impurities on dislocation lines, and accounts for the difficulty in resolving them at lower magnification. A \{100\} surface cleaved perpendicular to the outer surface of a crystal exhibiting such boundaries is shown in Fig. 19. It can be seen that pronounced kinks, which run back a short distance from the outer surface into the crystal, are associated with such regions.

The distribution of dislocations at small strains at 1000°C suggests a straightforward explanation for the observations described so far in this section. Let us assume that at 1300°C, as was proposed to be the case at 1000°C, slip is initiated first at surface sites, but that before slip bands develop to any great extent slip is also initiated by a number of segments of grown-in dislocation line throughout the volume of the crystal. At first, the primary slip bands develop very rapidly because all the instantaneous straining of the specimen is occurring at their edges. When secondary slip begins, however, their development is arrested because now most of the instantaneous shearing is associated with the secondary slip. The situation becomes an exaggeration of the previously described case of the specimen with many slip bands. If, as was the case at 1000°C, the primary slip bands provide resistance to secondary slip on the orthogonal slip plane, then edge dislocations will collect in their vicinity. Edge dislocations of opposite sign can climb together and annihilate each other. Edge dislocations of the same sign can lower the strain energy in such a region by polygonizing. Such polygonization boundaries would be pure tilt boundaries lying on the \{110\} planes perpendicular to the \{100\} surface on which the primary slip bands were initiated. They would, consequently, account for the dark lines observed in Fig. 17a and the cleavage markings observed
Fig. 18. Region near dark lines on the surface of the specimen shown in Fig. 17a at a higher magnification.
Fig. 19. Cleaved surface showing kink boundaries associated with dark lines in Fig. 17a.
in Fig. 19. That these boundaries occur only near the outer surface of the crystal suggests that at 1300°C screw dislocations may move faster than edge dislocations. If so, primary slip bands would be expected to form rapidly along the outer surface but would spread only slowly into the interior of the crystal. Thus, the onset of secondary slip would be likely to arrest the growth of many such bands near the surface at which they were initiated.

The failure of polygonization boundaries to form at the surface of the specimen deformed at 1600°C is probably due to a decrease in the resistance of the primary slip bands to the secondary slip. There is no stress interaction between dislocations moving on orthogonal slip planes in MgO because their Burgers vectors are at 90 deg. The resistance of the primary slip bands to the secondary slip must, then, result entirely from the intersection of primary and secondary slip dislocations. Any decrease in the density of dislocations in the primary slip bands decreases their resistance to secondary slip. It has already been pointed out that the dislocation dipole is the principal type of damage in slip bands of MgO. Washburn et al. have shown that at elevated temperatures dislocation dipoles are not stable, but pinch off into rows of prismatic loops. Such a process could, then, account for the postulated decrease in resistance of the primary slip bands.

If dislocation dipoles or some other type of damage do anneal out rapidly above about 1200°C, then the distribution of slip in specimens deformed at this temperature should be strain-rate sensitive. Figures 20a, b, and c show MgO single crystals deformed at stress rates of 2.6, 20, and 165 psi/sec as seen through crossed polaroids. Because of this stress rate difference, these specimens deformed plastically at different strain rates throughout loading. At 10,000 psi the strain rate of the 165-psi/sec specimen was $10^{-3}$ sec$^{-1}$, while that of the 2.6-psi/sec specimen was $10^{-4}$ sec$^{-1}$.

The distribution of birefringence in the 2.6-psi/sec specimen indicates that strain occurred about equally on both 90-deg slip systems. The same can be said for the 20-psi/sec specimen, except in this case the increase in birefringence and the obvious lattice rotation are
Fig. 20. Specimens strained about 15% at (a) 2.6 psi/sec, (b) 20 psi/sec, and (c) 165 psi/sec at 1300°C as seen with transmitted light through crossed polarizers.
evidence that intersection between the two 90-deg slip systems was more difficult. Lattice rotation is particularly evident at broad birefringence bands, indicating that many edge dislocations of one sign have collected in these regions.

In the specimen deformed at 165 psi/sec, it appears that intersection was still more difficult. A single crystal stressed along a \( \langle 100 \rangle \) axis must shear equally on orthogonal 90-deg slip systems if its ends are constrained. Normally this shearing occurs in the same region of a crystal, as happened in the 2.6- and 20-psi/sec specimens. At a stress rate of 165 psi/sec, however, intersection was apparently difficult enough so that a different shear distribution developed. Shearing on conjugate orthogonal slip systems took place at opposite ends of the specimen, thus extruding a section of crystal from its central region. The strain rate effect, therefore, supports the hypothesis that damage is being annealed out of dislocation bands at 1300°C. Increasing the stress rate increases the strain rate and thus decreases the amount of annealing time per increment of plastic strain.

Figure 21 shows a specimen deformed at 1600°C at a stress rate of 20 psi/sec. The decrease of birefringence and the absence of lattice rotation indicate that 90-deg intersections are no longer difficult. This is consistent with the fact that no polygonalization boundaries were observed on the surface of the specimen deformed at 1600°C shown in Fig. 17b. The tendency of pits to line up in \( \langle 100 \rangle \) directions on the surface of this specimen is probably due to interactions between dislocations on the oblique systems. Such interactions are discussed in detail in the next several paragraphs.

b. Interactions between dislocations on oblique slip systems

Figure 22 is a photograph of a \( \langle 100 \rangle \) oriented single crystal deformed to 14.8% strain at 1400°C. An interesting feature of this specimen is the vertical striations which appear on the upper part of the front face. Figure 23 shows a \( \langle 100 \rangle \) surface cleaved parallel to the front face of the crystal. The steps appearing on this surface show that dislocation boundaries are associated with these striations that run
Fig. 21. Specimen strained 15% at 1600°C as seen with transmitted light through crossed polarizers. The stress rate was 20 psi/sec.
Fig. 22. Specimen strained 14.8% at 1400°C as seen with transmitted light.
Fig. 23. Cleaved surface parallel to the front face, showing the deformation bands pictured in Fig. 22.
through the volume of the crystal. Figure 24 shows a \{100\} surface cleaved perpendicular to the front face of the crystal and also perpendicular to its stress axis. The steps on this surface indicate that the dislocation boundaries lie on \{110\} planes.

The striations and the associated dislocation boundaries are deformation bands resulting from the separation of regions that have deformed by slipping on planes at 45 deg to the crystal surface (bulging bands) from those that have slipped on planes at 90 deg (flat bands). At temperatures up to 1600°C, slip in a given region of a specimen deformed in this orientation was observed to occur on only two slip planes at 90 deg to each other. Slip on one such orthogonal set of slip systems apparently blocks slip on the other orthogonal set with the same resolved shear stress. It is thus indicated that even at the highest test temperatures the 60-deg intersections of the Burgers vectors are considerably more difficult than the 90-deg intersections.

Two types of dislocation boundary can be formed by dislocations moving on oblique \{110\} planes. The formation of these boundaries has previously been discussed by Kear, Taylor, and Pratt.

Dislocations moving on oblique planes with Burgers vectors that enclose an angle of 60 deg experience a repulsive force. Reactions of the type

$$\frac{a}{2} [\overline{1}01] + \frac{a}{2} [0\overline{1}4] = \frac{a}{2} [\overline{1}12]$$

do not occur because they cause an increase in elastic energy. A boundary may form, however, which can lie in any contact plane.

Dislocations moving on oblique planes with Burgers vectors that enclose an angle of 120 deg experience an attractive force. A reaction of the type

$$\frac{a}{2} [\overline{1}01] + \frac{a}{2} [0\overline{1}4] = \frac{a}{2} [\overline{1}10]$$

can occur to form segments of edge dislocation line lying along the \[[1\overline{1}1]\] direction. These segments can move only in the \{1\overline{1}2\} planes and are therefore sessile. A boundary can form which in \{101\} $\frac{a}{2} [\overline{1}01]$
Fig. 24. Deformation bands on surface cleaved perpendicular to surface of crystal shown in Fig. 22 and also perpendicular to its loading axis.
and $(011)^{\alpha} [011]$ dislocations lies on the $(\bar{1}10)$ plane and consists of $rac{a}{2} [\bar{1}10]$ edge dislocations lying one above the other and parallel to $[\bar{1}11]$. Each of the boundaries described above has a $[111]$ rotation axis as determined by the cross product $b_1 \times b_2$. Thus, the first boundary with a $(110)$ contact plane is a mixed tilt-twist boundary, while the second with a contact plane of $(\bar{1}10)$ is a pure tilt boundary. The mixed type of boundary has been reported by Taylor and Pratt$^{23}$ to form in squat compression specimens of NaCl and KCl. These boundaries are high energy boundaries, however, and should be rather diffuse, and it seems unlikely that they are responsible for the stable, sharp boundaries observed in our specimens.

The observed boundaries are thought to be the pure tilt boundaries. These boundaries have previously been observed to form in NaCl, LiF, and MgO bend specimens by Stokes, Johnston, and Li, who called them "anticlastic kinks."$^{24}$

Boundaries of this type remain sessile unless some complex dislocation movement takes place such as glide on $(112)$ planes. They attract or repel dislocations with a force which decreases gradually with temperature, because their only temperature dependence comes from the decrease in the shear modulus.

C. The Kinetics of the Plastic Deformation of MgO

The purpose of this section is to correlate observations of the distribution of dislocations in deformed MgO single crystals with the results of carefully controlled stress-strain measurements. Stress-strain results are analyzed by extending the dynamic approach, developed by Johnston and Gilman for explaining the yield behavior of LiF single crystals in a constant-displacement-rate test, to the constant-stress-rate method of testing used in this investigation.

1. The Relationship between Stress-Strain Behavior and Dislocation Behavior

As pointed out in Section III. A. 1, the slope of stress-strain curves obtained at a constant stress rate is related to the plastic strain rate by
The plastic strain rate can be related to dislocation behavior by

\[
\dot{\varepsilon} = m \rho b \bar{v} \quad (22)
\]

where \( \rho \) is the total length of mobile dislocation in the crystal divided by the crystal volume, \( b \) is the magnitude of the Burgers vector, \( \bar{v} \) is the average dislocation velocity, and \( m \) is a geometrical constant equal to 1/2 for single crystals of \( \text{MgO} \) stressed in a \( \langle 100 \rangle \) direction.

Johnston and Gilman's measurements of dislocation velocities in \( \text{LiF} \) and Johnston's measurements of dislocation velocities in \( \text{MgO} \) show that the velocity of screw and edge dislocations is related to the resolved shear stress by an equation of the form

\[
v = v_0 \left( \frac{\tau}{\tau_0} \right)^n \quad (23)
\]

where \( \tau \) is the resolved shear stress, \( v_0 \) equals 1 cm/sec, and \( \tau_0 \) and \( n \) are constants. Johnston and Gilman also found that in \( \text{LiF} \) edge dislocations travel at a substantially greater velocity than screw dislocations. Because of this velocity difference they show

\[
\bar{v} \approx 2v_s \quad (24)
\]

where \( v_s \) is the velocity of screw dislocations.

Equations (21) to (24) can be combined to give

\[
\frac{d\sigma}{dc} = \frac{1}{\rho b v_0 (\tau/\tau_0)^n} \frac{d\sigma}{dt} \quad (25)
\]

where \( m \) has been set equal to 1/2 and \( \tau_0 \) is the appropriate constant for screw dislocations. Thus, if the constants \( \tau_0 \) and \( n \), the shear stress acting on mobile dislocation line, and the variation of \( \rho \) during plastic deformation are known, the slope of the stress-strain curve at constant stress rate can be predicted.
2. The Kinetics of Plastic Deformation at Room Temperature

During the bulk yielding of MgO single crystals at room temperature, both the shear stress acting on a mobile dislocation line and the variation of \( \rho \) during plastic deformation are known. The shear stress acting on a dislocation moving through undeformed crystal is just the resolved shear stress calculated from the applied load. Also, because dislocation movement is restricted to slip band edges, \( \rho \) is proportional to the amount of interface area between slip bands and undeformed crystal.\(^7\)

Thus, for a specimen with a moderate number of slip band initiation sites, \( \rho \) should change rapidly during the period of slip band formation early in yielding when the strain rate is small and then remain essentially constant during yielding. If \( \tau \) in Eq. (25) is set equal to the resolved shear stress, \( \sigma/2 \), and \( \rho \) is held constant, Eq. (25) may be integrated to give directly the stress-strain curve at small strains,

\[
\epsilon = \frac{v_0 b}{n+4} \left( \rho \sigma_0^{-n} \right) \left( \frac{d \sigma}{dt} \right)^{-1} \sigma^{n+1} \tag{26}
\]

where \( \sigma_0 = 2\tau_0 \). If \( \rho \sigma_0^{-n} \) and \( n \) were known for the crystals used in this investigation, a direct comparison could be made between theoretical and experimental stress-strain curves. Because these parameters are not known, the consistency of Eq. (26) with experimental stress-strain curves was checked by determining whether it could reproduce such curves based on reasonable values for \( \rho \sigma_0^{-n} \) and \( n \).

Curve A in Fig. 25 was thus calculated from Eq. (26), with \( \rho \sigma_0^{-n} \) and \( n \) chosen so that curve A would match the experimental curve B. The best match was obtained by setting \( n \) equal to 33 and \( \rho \sigma_0^{-n} \) equal to \( 4.82 \times 10^{-13} \) cm\(^{-2}\) psi\(^{-33} \). This value of \( n \) is similar to values obtained by direct measurement of dislocation velocities in LiF and MgO.\(^2,3\) It will be shown that the value given for \( \rho \sigma_0^{-n} \) is also quite reasonable (a) by establishing an upper limit for \( \sigma_0 \) (assuming that \( n \) equals 33), and (b) by showing that the corresponding upper
Fig. 25. Experimental and calculated stress-strain curves at room temperature.
limit for \( p \), as determined by the condition that \( p \sigma_0^{-n} \) be equal to \( 4.82 \times 10^{-135} \text{cm}^{-2} \text{psi}^{-33} \), is consistent with the results of etch pit studies.

The distance, \( d \), that a dislocation moves during a time \( t \) can be obtained by integrating Eq. (23) (\( \tau \) and \( \tau_0 \) have been replaced by \( \sigma \) and \( \sigma_0 \)),

\[
d = v_0 \int_0^t \left( \frac{\sigma(t)}{\sigma_0} \right)^n dt .
\]  

An upper limit for \( \sigma \) can therefore be calculated on the basis of the fact that screw dislocation bands were observed in the gage sections of specimens deformed at room temperature at about 0.1\% strain. In the specimen whose stress-strain behavior is represented by curve B, a strain of 0.1\% corresponded to a compressive stress of about 14,800 psi. The stress rate for this specimen was 20 psi/sec and thus, at this stress, loading had been in progress for 740 seconds. The formation of screw dislocation bands could not have occurred unless screw dislocations had moved at least 3 mm. Thus, an upper limit can be set for \( \sigma_0 \) by making the appropriate substitutions in Eq. (27) and by carrying out the integration. The calculated upper limits for \( \sigma_0 \) and \( p \) are 16,640 psi and \( 9.47 \times 10^4 \text{cm}^{-2} \) respectively.

In Table I, a value of \( 1.7 \times 10^8 \) pits \text{cm}^{-2} \) was given for the density of etch pits in edge dislocation bands at room temperature. Edge dislocation bands were about 1 cm long in the compression specimens, and the total number of dislocation bands formed in the gage sections of specimens with a moderate amount of surface damage was about 200. If it is assumed that 1/10 of the etch pits lying within 0.1 \( \mu \) of slip-band edges corresponded to mobile dislocations, on the basis that (a) only dislocations lying close to slip-band edges can be mobile, and (b) most of the etch pits in edge dislocation bands correspond to dislocation dipoles; then, \( p \) should be about

\[
2 \times 200 \times 10^{-5} \times 1 \times 1.7 \times 10^8 \times 1/10 = 6.8 \times 10^4 \text{cm}^{-2} .
\]
This value is in reasonable agreement with the calculated upper limit, and thus it is concluded that the values for both $n$ and $\rho_0^{-R}$ used to "fit" curve B are consistent with the known experimental facts.

It is of particular interest that additional assumptions with regard to unpinning of dislocations or the sudden generation of mobile dislocation lines by dipoles are not necessary to explain the shape of the stress-strain curve at room temperature. It is determined by the stress dependence of the dislocation velocity. The agreement of the calculated and experimental curves suggests that slip band formation in the specimen occurred at strains less than 0.001.

The calculated and experimental curves in Fig. 25 begin to differ at a strain of 0.025. The behavior observed here is typical of specimens with few regions of surface damage. In this case, a relatively small number of slip bands forms and the spacing between these bands is large. Thus, substantial amounts of strain are necessary before slip band overlap begins to decrease the interface between strained and unstrained volumes of material.

Curve C in Fig. 25 is typical of specimens with many regions of surface damage. In this case, many slip bands with variable spacing are formed. It can be seen that increasing the number of regions of surface damage causes the strain rate to increase more rapidly with increasing stress in the yield region, but also causes strain hardening to occur at smaller strains.

The reason for the first effect is illustrated in Fig. 26. Most regions of surface damage are probably strain hardened. Thus, they would be expected to emit dislocations over a range of stresses. In this figure, $F(\sigma)$ represents the number of slip bands being initiated between $\sigma$ and $\sigma + d\sigma$. Thus, the total number of slip bands at a stress $\sigma$ is just

$$N = \int_{0}^{\sigma} F(\sigma) \, d\sigma$$  (27)
Fig. 26. Diagram illustrating how the number of slip band initiation sites may influence the shape of the stress-strain curve during yielding.
Here $F_1(\sigma)$ corresponds to a specimen with a large amount of surface damage, while $F_2(\sigma)$ corresponds to a specimen in which only a few slip bands can form.

Suppose $\sigma'$ is the stress at which the first detectable straining occurs in the specimen with many regions of surface damage. At this stress, the product

$$v(\sigma) \int_{0}^{\sigma'} F_1(\sigma) \, d\sigma$$

is proportional to the strain rate and therefore must equal some minimum detectable value. Obviously, a higher stress, $\sigma_2$, will be necessary to produce the same minimum detectable value in the specimen with only a few regions of surface damage. Once yielding begins, $\rho$ in the former specimen continues to increase, while $\rho$ in the latter one remains about constant. This is precisely the condition needed to explain the difference between curves B and C in the yield region.

The second effect is not so much a result of the large number of slip bands formed in the specimen with many regions of surface damage as it is of their nonuniform spacing. Slip bands originating from regions of extensive surface damage are closely spaced and thus overlap at smaller strains than slip bands with wider spacing. This overlap decreases $\rho$ because it decreases the amount of interface between deformed and undeformed volumes and thus causes hardening to occur. This mechanism was discussed previously in Section III. B. 1.

Figure 27 shows the calculated effect on stress-strain behavior of varying the stress rate. It also represents the effect of varying $\rho$ and keeping everything else constant. Increasing the loading rate or decreasing $\rho$ ($\rho$ is constant) shifts the stress-strain curve to higher stress values but does not change its shape. The magnitude of the effect, as shown in Fig. 27, agrees with the experimental results published by Hulse and Pask. The stress rate should affect the behavior of the specimen with many regions of surface damage. Higher stress rates will shift $\sigma_1$ to higher stress values, thus producing a shape typical of a specimen with few regions of surface damage. Such an effect can be seen in the curves of Hulse and Pask.
Fig. 27. Curves predicting the effect of stress rate variation at room temperature.
3. The Kinetics of Plastic Deformation at Elevated Temperatures

a. Stress-strain results

Stress-strain curves for specimens with \(\langle 100\rangle\) stress axis at temperatures from 1000°C to 1600°C are shown in Figs. 28-31. This orientation results in a maximum resolved shear stress on four of the six possible slip systems belonging to the \(\{110\}<11\overline{1}0\) family; the resolved shear stress vanishes on the other two slip systems as well as on the \(\{100\}<01\overline{1}\) family. The four consist of two pairs of orthogonal slip systems which intersect at 60 deg. Although the behavior of individual specimens agreed quite well up to 1300°C, some variation was observed for the different specimens tested at 1400°C. It was thought that this variation might be related to differences in impurity content between specimens. Thus, the stress-strain curves for 1300, 1510, and 1600°C, shown in Fig. 31 were obtained for single crystals cleaved from the same crystal block. Figure 32 shows that a slight decrease in yield stress occurs in the temperature range of 1000°C to 1600°C. The yield stress values reported here correspond to the stress supported by the specimens at 0.1% strain.

Of particular interest for temperatures above 1100°C is the change in the general shape of the stress-strain curves with the appearance of a gradual yield. This transition from an abrupt yield becomes more apparent at higher temperatures. Specimens were not normally deformed to failure at temperatures above 1100°C; they were often strained 15% without the appearance of cracks. At 1400°C one specimen was strained 30% without the appearance of a crack.

As a measure of strain-hardening rate, the slope of the first approximately linear portion of the stress-strain curve extending over an appreciable strain is plotted vs temperature in Fig. 33. At 1100°C and below it was observed that as the stress at a given strain decreased, this slope increased. Above 1100°C this slope decreased rapidly with increasing temperature. Data points at 1100°C and below in Fig. 33 are taken from the paper by Hulse and Pask.
Fig. 28. Stress-strain curves at 1000 to 1200°C for MgO single crystals compressed in a $\langle 100 \rangle$ direction at 20 psi/sec.
Fig. 29. Stress-strain curves at 1300°C for MgO single crystals compressed in a (100) direction at 20 psi/sec.
Fig. 30. Stress-strain curves at 1400°C for MgO single crystals compressed in a \((100)\) direction at 20 psi/sec.
Fig. 31. Stress-strain curves at 1300, 1510, and 1600°C for MgO single crystals cleaved from the same crystal block and compressed in a $\langle100\rangle$ direction at 20 psi/sec.
Fig. 32. The yield stress vs temperature curve for crystals with \(\langle 100\rangle\) and \(\langle 111\rangle\) stress axes compressed at 20 psi/sec. The yield stress at 560°C is 71,000 psi and at 360°C is 143,000 psi.
Fig. 33. Slope of the first linear part of the stress-strain curve vs temperature for (100) oriented single crystals.
Stress-strain curves for specimens with a (111) stress axis obtained at temperatures from 1200°C to 1600°C are shown in Fig. 34. This orientation results in a resolved shear stress on three of the six {100}(011) slip systems, and no stress on the {110}(110) family, as previously discussed. It is apparent that the strain hardening is considerably greater than that observed in the curves for the (100) oriented specimens. This result is undoubtedly due to the long-range repulsive stress interactions between dislocations arising because the Burgers vectors of the three active slip systems are at 60 deg to one another. As a consequence it was particularly difficult to constrain the sample ends from translating in planes perpendicular to the stress axis. In the 1500 and 1600°C specimens such translation resulted from slip taking place predominantly on one {100}(011) slip system, as was verified by examination of slip band birefringence with polarized light.

The yield stresses for (111) oriented specimens were considerably greater than those for the (100) orientations and continuously decreased up to 1600°C, as seen in Fig. 32. The ratio of the yield stresses is about 13:1 at 350°C, 4.5:1 at 600°C, and about 2.9:1 at 1600°C.

The effect of various stress rates on the plastic deformation of (100) oriented specimens at 1300°C is shown in Fig. 35. At a given strain, the slope of the stress-strain curve generally decreases with a decrease in the loading rate, as was the case at room temperature. The effect on the bulk yield stress was slight, although there was a decrease with decrease in loading rate. None of the specimens showed any fractures up to the test limit of 15% strain.

Figures 36 and 37 show the results of static recovery experiments carried out at 1000 and 1400°C for specimens compressed in the (100) direction with a stress rate of 20 psi/sec during the loading periods. The specimens were yielded, unloaded for a period of time, and then yielded again. This cycle was repeated several times. The stress-strain curve obtained at 1000°C, following this procedure, is particularly interesting because the abrupt yielding followed by a high
Fig. 34. Stress-strain curves for MgO single crystals compressed with a (111) stress axis at various temperatures. All specimens were loaded at 20 psi/sec.
Fig. 35. Stress-strain curves for MgO single crystals compressed with a (100) stress axis at various constant stress rates. The specimens deformed at 165, 80, and 22 psi/sec were all cleaved from the same crystal block.
Fig. 36. Static recovery experiment on MgO single crystal at 1000°C. Specimen loaded at 20 psi/sec.
Recovery experiment
T = 1400 °C

Fig. 37. Static recovery experiment on MgO single crystals at 1400°C. Specimen loaded at 20 psi/sec.
strain rate indicates that a pinning of dislocations, probably by diffusion of impurity atoms to them, occurred while the specimen was unloaded. This result is consistent with previous observations by Elkington et al.\textsuperscript{12} and also by May and Kronberg\textsuperscript{11} of the appearance of a yield point for MgO deformed above 750°C.

Some idea of the rate of pinning can be obtained by comparing the results of unloading the specimen for various lengths of time. Little pinning was observed after unloading the specimen for 2 minutes, but the 5-minute unloading produced as much pinning as the 30-minute one.

At 1400°C the static recovery behavior of MgO is quite similar to that of a metal deformed at a temperature exceeding half its melting point. Much of the dislocation damage introduced during plastic strain- anneals out during the unloaded periods. This annealing out appears to occur quite rapidly. The term "orthorecovery" has often been used to describe this behavior in metals.

b. Yielding kinetics

No measurements have yet been reported of the temperature variation of average dislocation velocities in MgO. Such measurements have been made, however, for LiF over a relatively narrow temperature range, and have shown that increasing the temperature from 77 to 300°K decreased \( \tau_0 \) while \( n \) remained about constant.\textsuperscript{2} Taking into consideration the relative melting points of LiF and MgO, this temperature range is equivalent to -71 to 531°C in MgO.

Curve A in Fig. 38 was calculated with the same values of \( \rho \) and \( n \) as curve A in Fig. 25, but with \( \tau_0 \) adjusted to give the same yield stress as curve B, which is the 1000°C curve in Fig. 28. A feature of the experimental curve B which is predicted by Eq. (26) is the sharpness of the yield. It differs from the calculated curve, however, because strain hardening is occurring at small strains. This suggests that dislocation interactions exist at small strains at 1000°C which do not occur at room temperature. The values of \( \rho \), \( n \), and \( T \) for all the calculated curves in Fig. 38 are given in Table II.
Fig. 38. Calculated and experimental stress-strain curves for MgO single crystals deformed under various conditions at elevated temperatures.
Table II. Parameters used to calculate theoretical stress-strain curves [see Eq. (26) and Fig. 38].

<table>
<thead>
<tr>
<th>Curve</th>
<th>( T ) (°C)</th>
<th>( \dot{\sigma} ) (psi/sec)</th>
<th>( n )</th>
<th>( \sigma_0 ) (psi)</th>
<th>( \rho ) (cm/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1000</td>
<td>20</td>
<td>33</td>
<td>4580</td>
<td>( 9.47 \times 10^4 )</td>
</tr>
<tr>
<td>B</td>
<td>1000</td>
<td>20</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>1300</td>
<td>20</td>
<td>2.08</td>
<td>142200</td>
<td>( 9.47 \times 10^4 )</td>
</tr>
<tr>
<td>D</td>
<td>1300</td>
<td>20</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E</td>
<td>1300</td>
<td>80</td>
<td>2.08</td>
<td>142200</td>
<td>( 9.47 \times 10^4 )</td>
</tr>
<tr>
<td></td>
<td>1300</td>
<td>20</td>
<td>2.08</td>
<td>142200</td>
<td>( 9.47 \times 10^4 )</td>
</tr>
<tr>
<td></td>
<td>1300</td>
<td>2</td>
<td>2.08</td>
<td>142200</td>
<td>( 9.47 \times 10^4 )</td>
</tr>
</tbody>
</table>
On the basis of the dominant role of dislocation mobility in determining the shape of the stress-strain curve at room temperature and the temperature dependence of dislocation mobility observed in LiF, the most straightforward explanation for the decrease in yield stress for ⟨100⟩ stressed specimens with increasing temperature would appear to be increased dislocation mobility. A difference in dislocation mobility is also probably responsible for the difference in yield stress between specimens stressed in ⟨100⟩ and ⟨111⟩ directions. When a dislocation moves on a {110} plane, ions of opposite sign approach each other at the core of the dislocation in a "midglide" position. For dislocation movement on {100} planes, on the other hand, Gilman has pointed out that strong repulsive forces develop because ions of like sign are forced to approach each other. 25 Because of this structural reason for the difference in dislocation mobility on {100} and {110} planes, it seems unlikely that the bulk yield stress of ionic single crystals with the NaCl structure should be the same for both slip systems at temperatures up to the melting point.

Curve C in Fig. 38 is a curve calculated by using Eq. (26) with \( \tau_0, \rho, \) and \( n \) chosen to match curve D, which is the 1300°C curve from Fig. 31. In this case, the best fit was obtained by choosing \( n \) to be about 2. Thus, at 1300°C the strain rate is much less sensitive to stress than at 1000°C. Shapes similar to that shown in curve C were observed for all specimens tested above 1100°C.

It is likely that the decrease in stress sensitivity of the strain rate starting at 1100°C is due to a decrease in stress sensitivity of the dislocation velocity. Such a decrease might occur, for example, if dislocation motion above 1100°C was limited by the nonconservative motion of jogs. It is doubtful, however, that the values of \( n \) and \( \tau_0 \), or, for that matter, Eq. (23) - represent the dislocation velocity above 1100°C because there is evidence that Eq. (26) is no longer valid.

Equation (26), for example, does not correctly predict the effect of varying the stress rate at 1300°C as it does at room temperature. This condition becomes apparent by comparing curves E-80 and E-2 Fig. 38 (which are calculated by using the same values of \( \rho, \tau_0 \) and \( n \).
as curve C—the same as curve E-20 with the experimental curves in Fig. 35. Although the calculated curves predict qualitatively the correct behavior, the agreement with respect to the magnitude of the stress rate effect is poor.

Thus, it appears that at 1300°C one or more of the assumptions used to derive Eq. (26) no longer apply. The dislocation velocity may no longer be given by Eq. (23). The shear stress acting on moving dislocations may no longer be that calculated from the applied load due to interactions between dislocations arising from a change in the distribution of dislocations at small strains. The parameter \( \rho \) may no longer be constant in the yield region. A decrease in the stress dependence of the dislocation velocity would cause \( \rho \) to play a greater part in determining the shape of the stress-strain curve during yielding.

c. Strain hardening

A material is said to strain harden if its yield stress increases upon reloading after an increment of plastic strain; the slope of a stress-strain curve is thus often taken as a measure of strain-hardening rate. In making such an interpretation, however, it must be realized that the resistance of a test specimen to dislocation motion may vary considerably from region to region and that the slope of the stress-strain curve at a given point measures only the resistance to dislocation motion in the region where such resistance is least.

For a given applied load, strain hardening results from a decrease of the net shear stress acting on moving dislocations caused by straining of the crystal. Such a decrease occurs in a deformed volume of material due to stress interactions between the moving dislocations and other dislocations and defects present in the volume. It can also occur in a compression specimen as a result of lattice rotation relative to the compression axis.

A decrease of the net shear stress acting on moving dislocations causes them to move more slowly or to stop moving altogether. Thus, the length of moving dislocation line, the average dislocation velocity, and consequently the strain rate all decrease for a given applied load.
Strain hardening at room temperature has already been discussed. At small strains, it is due to the decrease in interface area between deformed and undeformed volumes of material, and is sensitive to the number and distribution of slip bands in the crystal. At large strains ($\varepsilon > 0.0425$) further straining requires that dislocations move through deformed regions. Specimens always begin fracture before this strain is reached.

Strain hardening at $1000^\circ C$ is greater, by a factor of 5, than that at room temperature. The distribution of dislocations in specimens deformed at $1000^\circ C$ (as described in Section III. B. 3) suggests that this increase is related to the initiation of secondary slip at the grown-in substructure. In this case the dislocation density increases uniformly throughout the volume of the crystal and thus there are no undeformed regions in which dislocation movement can take place. At exactly what temperature the grown-in substructure becomes mobile is not known. The data of Hulse and Pask indicate that it may occur as low as $800^\circ C$.  

Beyond $1100^\circ C$, $d\sigma/d\varepsilon$ begins to decrease rapidly with increasing temperature and by $1600^\circ C$ has decreased by a factor of about 8. This decrease correlates with the disappearance of polygonalization boundaries as described in Section III. B.3. The disappearance of these boundaries was attributed to the annealing-out of dislocation dipoles, and it was shown that the distribution of slip at $1300^\circ C$ was strain-rate sensitive, as would be expected if some of the dislocation damage produced during deformation could be annealed out in time. The annealing out of the dislocation dipole probably accounts for much of the recovery observed in the static recovery experiment at $1400^\circ C$.

The static recovery experiment at $1400^\circ C$ indicates that a part of the dislocation damage produced at $1400^\circ C$ is also thermally stable. At each successive loading, yielding occurred at a higher stress. The results of the stress rate experiments shown in Fig. 35 also indicate that thermally stable dislocation damage is produced which accumulates with increasing strain. This effect can be seen by comparing the strain rates of the various constant-stress-rate curves at 10,000 psi. These strain rates are listed in Table III. At 10,000 psi the smallest strain
<table>
<thead>
<tr>
<th>$\frac{d\sigma}{dt}$ (psi/sec)</th>
<th>$\frac{d\sigma}{d\epsilon}$ (psi)</th>
<th>$\frac{d\epsilon}{dt}$ (sec$^{-1}$)</th>
<th>$\epsilon$</th>
</tr>
</thead>
<tbody>
<tr>
<td>165</td>
<td>$1.8 \times 10^5$</td>
<td>$0.9 \times 10^{-3}$</td>
<td>0.032</td>
</tr>
<tr>
<td>80</td>
<td>$8.0 \times 10^4$</td>
<td>$1.0 \times 10^{-3}$</td>
<td>0.038</td>
</tr>
<tr>
<td>20</td>
<td>$5.0 \times 10^4$</td>
<td>$4.4 \times 10^{-4}$</td>
<td>0.060</td>
</tr>
<tr>
<td>2</td>
<td>$2.0 \times 10^4$</td>
<td>$1.0 \times 10^{-4}$</td>
<td>0.080</td>
</tr>
</tbody>
</table>
rate was observed in the case of the 2-psi/sec specimen because this specimen had strained the most in spite of the fact that the amount of time for annealing out of damage per increment of strain was greatest in this specimen. One type of damage which is known to be stable at elevated temperatures is that associated with the deformation bands discussed in Section III. B. 3. Interactions between dislocations on oblique slip planes probably account for most of the thermally stable damage.
IV. SUMMARY

An apparatus was designed and built for making carefully controlled stress-strain measurements at temperatures up to 1600°C. Compression loading was used, rather than bend or tension, for two reasons: (a) The stress distribution in an elastically compressed right prism (the shape of specimens used in this investigation) is both uniaxial and uniform; and (b) no special specimen grips are required for compression loading, as would be required for tensile loading. Strain was measured in a central gage section and the load was applied at a constant stress rate, rather than at a constant crosshead displacement rate, in order to minimize complications arising from a nonuniform distribution of shear strain. A positive constraint was applied to specimen ends in order to keep them from translating over the parallel surfaces of the loading ram during deformation. A complete discussion of this type of stress-strain measurement was given in Sections II.A.1, 2, and 3.

Stress-strain measurements were obtained for MgO single crystals compressed with (100) and (111) stress axes at temperatures ranging from 1000 to 1600°C. These were correlated with etch pit and stress birefringence studies of the distribution of dislocations in single crystals at various stages of deformation. Similar correlations were also made in the temperature range 26 to 1000°C, where the stress-strain behavior of MgO had already been established by previous investigations.

A. (100) Specimens

Specimens with (100) stress axes deform plastically by slip on (110)〈110〉 slip systems. At room temperature, the shape of the stress-strain curve is determined by the dislocation mobility and the number of slip bands formed. Dislocations move only in undeformed regions at slip band edges. Strain hardening results from slip band overlap.

Between 26 and 1000°C, the dislocation mobility increases with increasing temperature, thus causing the yield stress to decrease. The etch pit density in slip bands formed at 500°C is less than that at
room temperature, while the total shear strain is about the same. At 1000°C, slip bands still form; however, dislocations were also found to be distributed throughout the volume of the crystal. This effect was attributed to the initiation of slip by grown-in dislocations. The slope of the stress-strain curve, after yielding, is about five times as great at 1000°C as at room temperature because there are now no undeformed regions in which dislocations can move.

Above 1100°C, the yield stress decreases slowly with increasing temperature and the strain rate becomes much less stress-sensitive. The strain-hardening rate decreases rapidly owing to the annealing out of thermally unstable dislocation damage such as dislocation dipoles. At 1300°C, polygonalization walls, and dislocation boundaries formed by dislocations lying on oblique slip planes, obstruct dislocation movement. Above 1400°C, polygonalization walls were no longer observed to form.

B. \{111\} Specimens

Specimens with \{111\} stress axes deform plastically by slip on \{100\}\{011\} slip systems. The ratio of yield stresses of \{111\} specimens to \{100\} specimens decreases rapidly from 1000 to 1600°C. Strain hardening for \{111\} specimens was much higher than for \{100\} specimens: The Burgers vector of the active slip systems lie at 60 deg (or 120 deg) to each other and thus there are long-range stresses between intersecting dislocations.
The writer wishes to thank Professor Joseph A. Pask, who was the thesis supervisor, and Professor Jack Washburn for their advice and encouragement throughout the course of this investigation.

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APPENDIX A

The interaction of a specimen with a constant crosshead displacement testing machine is shown schematically in Fig. A-1. Johnston and Gilman have pointed out that in such a test, the crosshead motion deforms the machine as well as the specimen, so that

\[ RT = \Delta S + \Delta L_E + \Delta L_P, \quad (A-1) \]

where \( \Delta S \) is the part of the crosshead motion going to elastic strain of the apparatus and \( \Delta L_E \) and \( \Delta L_P \) are the parts going to the elastic and plastic strains of the specimen.

If Eq. (A-1) is divided by \( L_0 \) and differentiated with respect to time, and \( \Delta S \) and \( \Delta L_E \) are eliminated by being expressed in terms of stress, one obtains

\[ \frac{d\varepsilon_N}{dt} - \frac{d\varepsilon_p}{dt} = \frac{1}{E} \frac{d\sigma}{dt} \left\{ \frac{AE}{L_0 K} + 1 \right\}, \quad (A-2) \]

which can be written

\[ \frac{d\sigma}{dt} = \frac{L_0 K}{A} \left\{ \frac{d\varepsilon_N}{dt} - \frac{d\varepsilon_p}{dt} \right\}, \quad (A-3) \]

provided that

\[ \frac{AE}{L_0 K} \gg 1, \quad (A-4) \]

i.e., provided that the elastic strain stored in the apparatus is much greater than that stored in the specimen.

The stress rate can be expressed in terms of the strain rate using the chain rule:

\[ \frac{d\sigma}{dt} = \frac{d\sigma}{d\varepsilon_N} \frac{d\varepsilon_N}{dt}. \quad (A-5) \]
**Fig. A-1.** A schematic representation of a constant crosshead displacement apparatus. The crosshead moves at constant speed $R$, compressing the crystal. Part of the crosshead motion goes to the elastic strain of the machine; it is represented by an imaginary spring with force constant $K$. The displacement rate is given by $R/L_0 = \dot{\varepsilon}_N$, where $R$ is the displacement rate, $L_0$ is the sample height, $\dot{\varepsilon}_N$ is the strain rate, and $K$ is the force constant. Additionally, $A$ is the cross section, $E$ is Young's modulus, and $\mu$ is a coefficient.
By substituting this equation into (A-3), one obtains

\[
\frac{d\sigma}{d\epsilon_N} = \frac{L_0 K}{A} \left\{ 1 - \frac{d\epsilon_p}{dt} \left( \frac{d\epsilon_N}{dt} \right)^{-1} \right\}.
\]

(A-6)

Similarly, it can be shown that

\[
\frac{d\sigma}{d\epsilon_p} = \frac{L_0 K}{A} \left\{ \frac{d\epsilon_N}{dt} \left( \frac{d\epsilon_p}{dt} \right)^{-1} \right\}.
\]

(A-7)

Equations (A-3), (A-6), and (A-7) are the same as Eqs. (6), (4), and (5) in Section III.
REFERENCES


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