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DEFORMATION OF POLYCRYSTALLINE CERAMICS

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Conf. on Role of Grain Boundaries and Surfaces in Ceramics, N. Carolina State of the U. of N. Carolina at Raleigh, Nov. 16-18, 1964.

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DEFORMATION OF POLYCRYSTALLINE CERAMICS

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April 5, 1965

DEFORMATION OF POLYCRYSTALLINE CERAMICS*

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ABSTRACT

The mechanical behavior of nominally dense, pure, polycrystalline, ceramic materials in which dislocations are mobile is discussed. Deformation by crystallographic slip is first considered. The von Mises analysis is developed for a two-dimensional polycrystalline aggregate, and a generalization is then made to three dimensions and to simple ceramic structures. Deformation by grain boundary sliding is also considered. Stress-strain curves and photomicrographs are presented for two types of polycrystalline MgO at temperatures up to 1500° C that illustrate both deformation modes. Problems encountered in realizing deformation of polycrystalline ceramic materials are reviewed, and methods for modifying the behavior of ceramic materials in which dislocations are mobile are discussed.

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*Presented at the Conference on Role of Grain Boundaries and Surfaces in Ceramics, North Carolina State of the University of North Carolina at Raleigh, November 16-18, 1964.

I. INTRODUCTION

If a polycrystalline material is to exhibit appreciable ductility, it must deform primarily by crystallographic slip. Each grain must contain segments of dislocation line that can move and multiply, thereby spreading slip throughout its volume. To maintain continuity of material during deformation, each grain must also be able to undergo general changes in shape. This is possible, however, only if slip occurs on certain families of slip systems. Finally, for ductility, the grain boundaries must be strong enough to transmit stresses between grains without shearing or parting.

Although a number of ceramic materials exhibit ductility as single crystals, polycrystalline ceramics are generally brittle. In this report it is shown that such behavior can generally be attributed to failure to satisfy one or more of the preceding conditions. The discussion is restricted to dense, single-phase, polycrystalline materials. Section II deals with crystallographic slip; Section III, with grain boundary sliding; and Section IV, with experimental data on polycrystalline MgO.

II. CRYSTALLOGRAPHIC SLIP

A. The von Misés Analysis

Slip produces a displacement parallel to certain crystallographic planes and directions. Each slip plane and corresponding slip direction is called a slip system. In metals, the slip plane is generally that of densest packing and widest spacing; the slip direction, that of the densest row of atoms. In ceramic materials, the choice of slip plane and slip direction is also affected by the presence of electrostatic forces and directional bonds.

To be considered is a volume element in a polycrystalline material which is small enough so that the distribution of shear is approximately uniform and yet large enough so that the inhomogeneities inherent in slip itself are not resolved. The displacement $\{u_i\}$ of an arbitrary point $\{x_i\}$ due to a shear strain $d\gamma$ parallel to the $\overline{12}$ plane and the 2 direction is shown in Fig. 1. The magnitude of the displacement is given by the product of the shear strain and the perpendicular distance from the point to the slip plane containing the origin. The direction of the displacement is the shear direction. The ith component of the displacement vector can be obtained by multiplying its magnitude by the cosine of the angle between the slip direction and the ith axis. Thus, the displacement of a point due to a uniform shear parallel to a plane whose normal is $\{n_i^\alpha\}$ and in a direction $\{\ell_i^\alpha\}$ is given by

$$u_i^\alpha = \sum_{j=1}^{3} d\gamma^\alpha \, n_j^\alpha \, x_j \, \ell_i^\alpha , \tag{1}$$

where the index α is used to label slip systems. The total displacement of a point due to shear on n slip system is

$$u_i = \sum_{\alpha=1}^{n} u_i^\alpha . \tag{2}$$

The strain components can be obtained from the total displacement by differentiating with respect to position coordinates.

$$\epsilon_{ij} = \frac{1}{2} \left(\frac{\delta u_i}{\delta x_j} + \frac{\delta u_j}{\delta x_i} \right) \tag{3}$$

Since $\epsilon_{ij} = \epsilon_{ji}$, Eq. (3) represents a set of six linear equations relating the strain components to the shears. In shear deformation, no

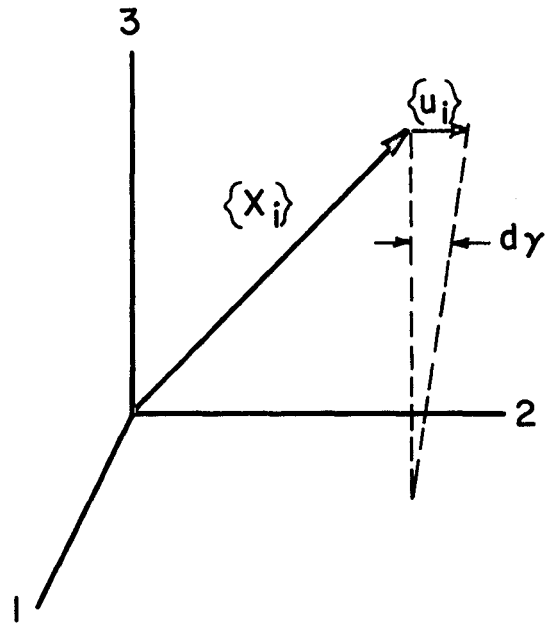

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Fig. 1. The displacement of a point resulting from shear parallel to the (001) plane in the [010] direction, i.e., $\{n_i\} = (0, 0, 1)$ and $\{\ell_i\} = (0, 1, 0)$.

volume change occurs and thus $\epsilon_{11} + \epsilon_{22} + \epsilon_{33} = 0$. It follows that the most general uniform strain that can be produced by shear deformation can be represented by five independent strain components. The following cases result by omitting one of the equations for the normal strains and setting E equal to the matrix of coefficients of $d\gamma^{\alpha}$:

1. If the number of shears is less than five ($n < 5$), it is not possible to fix the five strain components independently.
2. If $n = 5$, it is possible to fix the five strain components independently providing that the determinant of $E \neq 0$.
3. If $n > 5$, it is possible to fix the five strain components independently providing that a 5 x 5 minor of $E \neq 0$. In this case, $n - 5$ shears may be assigned arbitrary values.

Thus, five shears, independent in the sense that they can independently fix five components of the strain tensor, are required to produce the most general uniform strain. This result was first derived by von Misés.[1] The analysis here follows that of Bishop.[2] Although it is valid only for infinitesimally small strains, this analysis can be applied to large strains by viewing the components of the strain tensor as strain rates multiplied by an infinitesimal length of time.

Because of Taylor's analysis, it has generally been assumed that a polycrystalline material could maintain continuity during plastic deformation only if its grains could undergo a general uniform strain.[3] On the basis of the von Misés analysis, this condition is equivalent to requiring that a material have five independent slip systems. A clearer

picture of why slip systems are independent or dependent and why a certain number of slip systems is necessary to maintain continuity during deformation can be obtained by considering a two-dimensional polycrystalline material.

B. Deformation of a Two-Dimensional Polycrystalline Aggregate

In two dimensions, there are three strain components ϵ_{11}, ϵ_{22}, and ϵ_{12}. Of these, however, only two can be independently fixed because area is conserved in shear deformation so that $\epsilon_{11} + \epsilon_{22} = 0$. Consequently, only two independent slip directions are required for a general change in shape.

Figure 2 shows a two-dimensional crystal containing three slip directions, [10], [11], and [01]. The total displacement of a point due to shear parallel to these slip directions may be calculated from Eqs. (1) and (2) with the appropriate values of $\{n_i^\alpha\}$ and $\{\ell_i^\alpha\}$ as indicated below.

α	slip system	n_1	n_2	ℓ_1	ℓ_2
1	[10]	0	1	1	0
2	[11]	$-1/\sqrt{2}$	$1/\sqrt{2}$	$1/\sqrt{2}$	$1/\sqrt{2}$
3	[01]	1	0	0	1

Substituting and regrouping, the total displacements become

$$u_1 = -\frac{1}{2}\, d\gamma^2\, x_1 + (d\gamma^1 + \frac{1}{2}\, d\gamma^2)x_2 \tag{4a}$$

$$u_2 = (-\frac{1}{2}\, d\gamma^2 + d\gamma^3)x_1 + \frac{1}{2}\, d\gamma^2\, x_2 \,. \tag{4b}$$

Differentiating the total displacements with respect to position

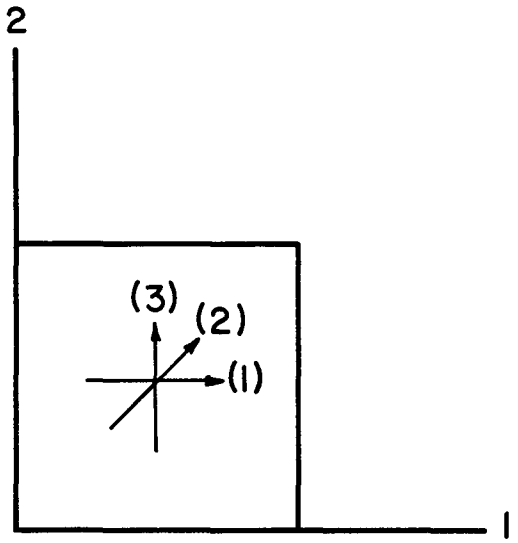

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Fig. 2. A grain with [10], [11], and [01] slip directions.

coordinates, as in Eq. (3), the following strain components are obtained:

$$\epsilon_{11} = -\frac{1}{2}\, d\gamma^2 \tag{5a}$$

$$\epsilon_{22} = \frac{1}{2}\, d\gamma^2 \tag{5b}$$

$$\epsilon_{12} = \epsilon_{21} = \frac{1}{2}\, d\gamma^1 + \frac{1}{2}\, d\gamma^3 \,. \tag{5c}$$

By omitting one of the normal strain equations (Eq. 5a), an E-matrix with a nonvanishing 2 x 2 minor is obtained. Thus, slip directions 1, 2, and 3 constitute two independent slip directions. It can be seen that directions 1 and 2 or 2 and 3 also constitute two independent slip directions, but directions 1 and 3 provide only one.

In physical terms, a slip system (or in two dimensions a slip direction) is independent if shear on it produces a change in shape which cannot be produced by a superposition of shears on the other slip systems. Slip systems fail to be independent if, because of crystal symmetry, shear on one produces a change in shape which differs by only a rigid body rotation from that produced by shear on the other. In two dimensions, orthogonal slip systems such as 1 and 3, shown in Fig. 3, provide a good example of this case. Slip parallel to [10] produces a shape that differs by only a 90° rotation from the shape produced by slip parallel to $[0\bar{1}]$. Another case in which slip systems fail to be independent is found in fcc metals where slip occurs on the {111} $\langle 1\bar{1}0 \rangle$ slip systems. Although each slip plane contains three slip directions, only two of the resulting three slip systems are independent: a change in shape due to shearing on one slip system can always be produced by a superposition of shearing parallel to the other two.

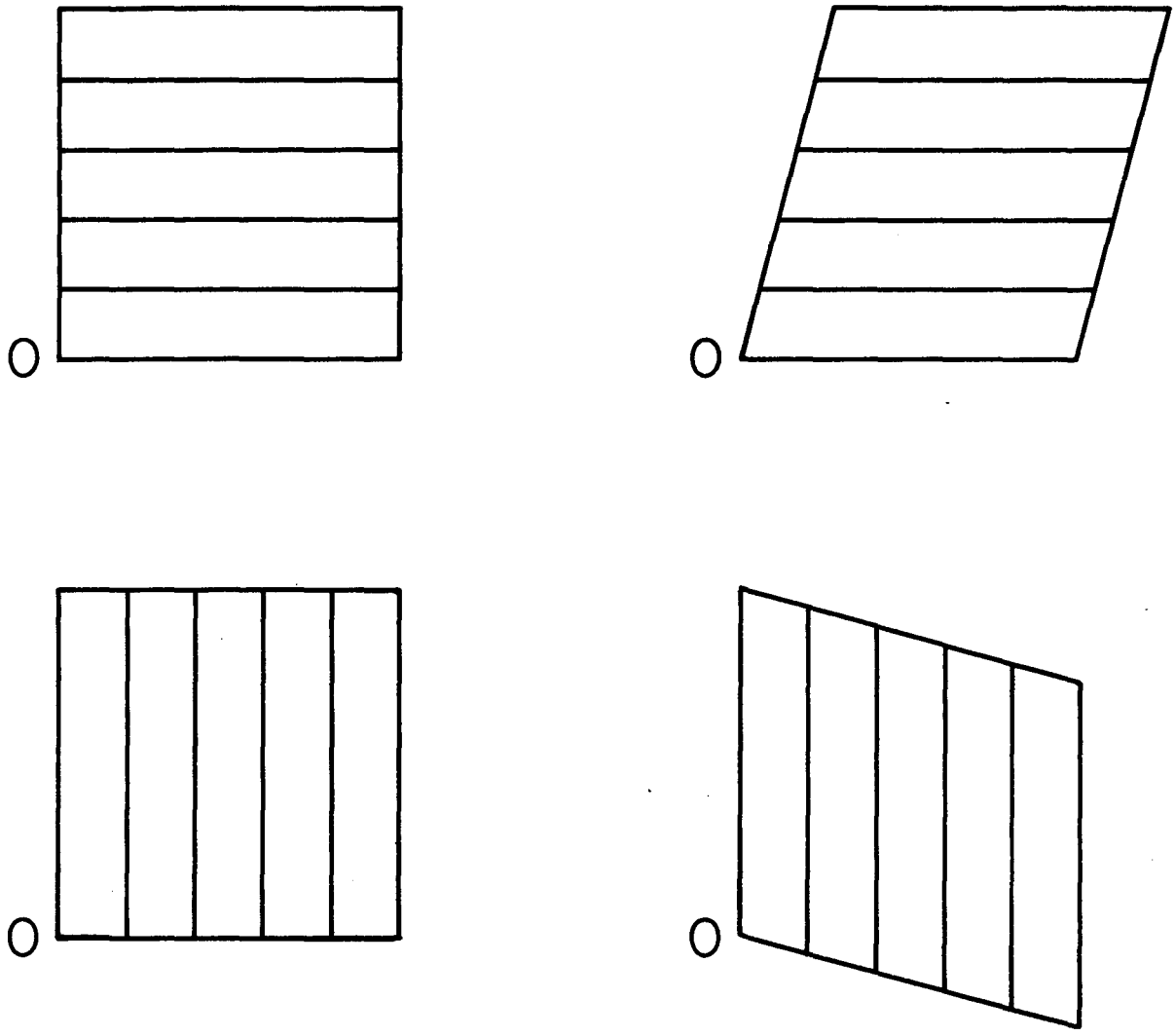

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Fig. 3. The change in shape of a two-dimensional grain resulting from shear parallel to two orthogonal slip directions.

Figure 4a shows the grains of a two-dimensional polycrystalline specimen, before and after uniaxial compression. Continuity is maintained because each grain has taken on the overall strain of the specimen (i.e., in each grain ϵ_{22} equals the compressive strain of the specimen, $\epsilon_{11} = -\epsilon_{22}$ to conserve area and $\epsilon_{12} = 0$, where the strain components are referred to axes parallel and perpendicular to the compression axis). A grain oriented at random with respect to the compression axis can change shape in the required manner, however, only if it has two independent slip directions. The possible shape changes for a grain with two orthogonal slip directions and therefore only one independent slip direction are shown in Figs. 4b, c, and d. The required change of shape can only be obtained if the grain is oriented with its slip systems at 45° to the compression axis as shown in Fig. 4b. In this orientation, shear parallel to one slip direction produces the required change of shape accompanied by a rotation. Equal amounts of shear parallel to both slip directions produce the same change of shape with no rotation. Grains in other orientations, however, cannot change shape as required. The grain in Fig. 4c can take on the required uniaxial strain ϵ_{22} but will not remain rectangular regardless of how shear is distributed between its two slip directions. The grain in Fig. 4d cannot strain along the specimen axis. Thus, it can be seen that continuity cannot be maintained in a polycrystalline material with randomly oriented grains if deformation occurs on an insufficient number of independent slip systems.

C. Application of the von Misés Analysis to Ceramic Structures

Groves and Kelly have recently examined a number of simple ceramic structures to see if they possess the five independent slip systems that

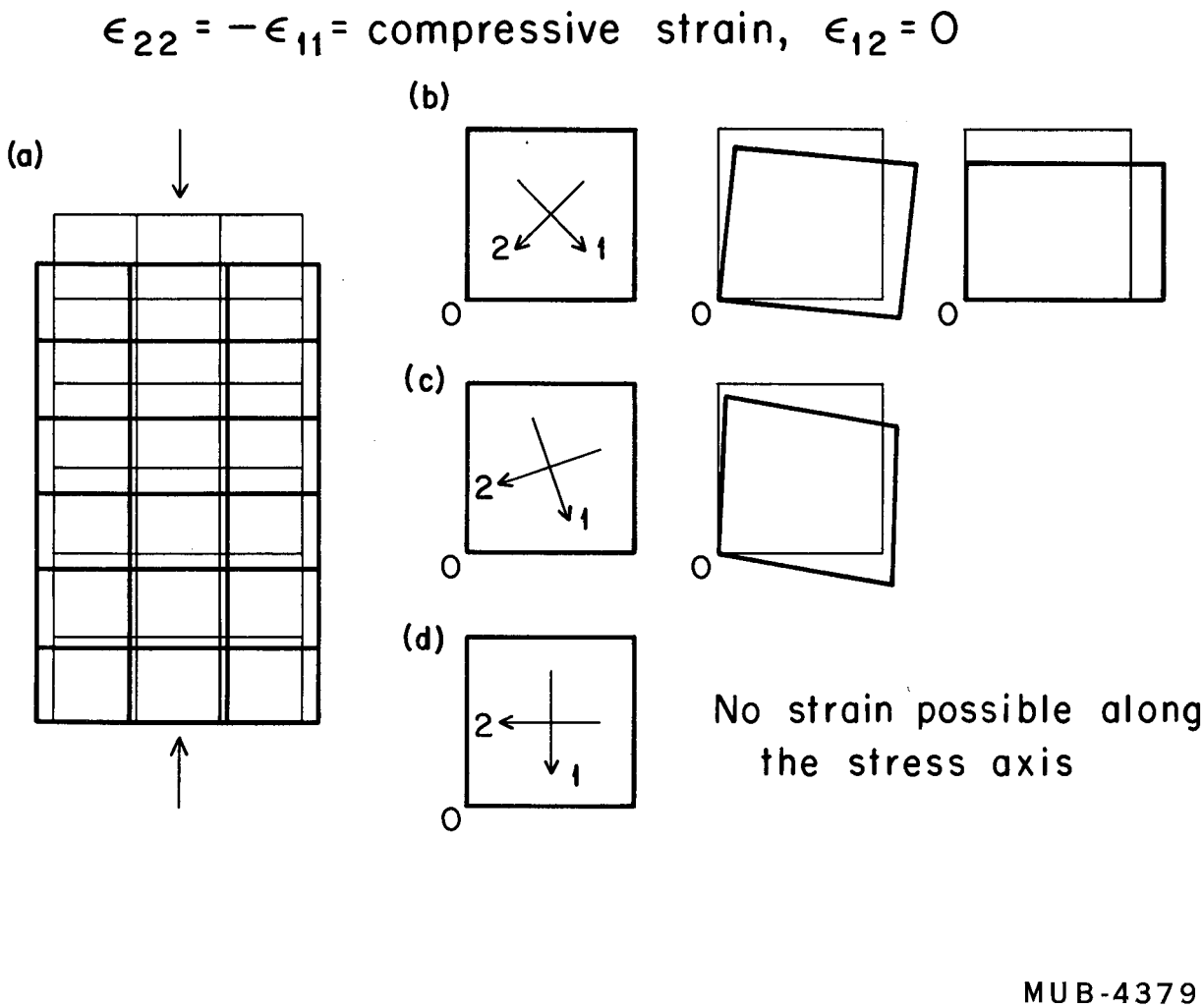

Fig. 4. The possible changes of shape of a two-dimensional grain with only one independent slip system.

are required for polycrystalline ductility.[4] Their results are summarized in Table I.

In crystals with the NaCl structure, slip can occur on two families of slip systems. In those crystals with ionic bonding, at low temperatures, dislocations move on {110} $\langle 1\bar{1}0 \rangle$ slip systems producing straight slip steps; at somewhat higher temperatures dislocations can also move on {001} $\langle 1\bar{1}0 \rangle$ slip systems producing slip steps that are wavy.[5] According to Table I, slip must occur on both the {110} $\langle 1\bar{1}0 \rangle$ and the {001} $\langle 1\bar{1}0 \rangle$ families to provide five independent slip systems; the {110} $\langle 1\bar{1}0 \rangle$ family alone provides only two. As predicted, polycrystalline NaCl,[6] AgCl,[7] KCl,[8] and LiF[9] are brittle at low temperature but become ductile at temperatures where slip on both {110} $\langle 1\bar{1}0 \rangle$ and {001} $\langle 1\bar{1}0 \rangle$ families is possible. Similar behavior has been observed by the authors in polycrystalline MgO and will be described in Section IV.

In alumina, slip occurs on (0001) $\langle 11\bar{2}0 \rangle$; however, there is a lack of agreement as to the second slip system.[4,10] Groves and Kelly, using the (0001) $\langle 11\bar{2}0 \rangle$ and $\{10\bar{1}0\}$ $\langle 11\bar{2}0 \rangle$ slip systems, showed that they provided only four independent slip systems. Although single crystals of sapphire deform in tension above 1270°C (the transition temperature is strain rate sensitive),[11] polycrystalline Al_2O_3 in the form of "Lucalox" exhibits no ductility up to 1900°C.[12] In addition to its lack of sufficient independent slip systems, the ductility of polycrystalline Al_2O_3 is also limited at low temperatures by a high frictional resistance to dislocation motion. CsCl, on the other hand, has a low resistance to dislocation motion at room temperature and thus is extremely soft. The {110} <001> slip systems provide only three independent slip systems,[13]

Table I. Families of slip systems for simple ceramic structures

Structure	Slip systems	Number of independent systems
NaCl	$\{110\}\ \langle 1\bar{1}0\rangle$	2
	$\{110\}\ \langle 1\bar{1}0\rangle + \{001\}\ \langle 1\bar{1}0\rangle$	5
Al_2O_3	$(0001)\ \langle 11\bar{2}0\rangle$	2
	$(0001)\ \langle 11\bar{2}0\rangle + \{10\bar{1}0\}\ \langle 11\bar{2}0\rangle$	4
CsCl	$\{110\}\ \langle 001\rangle$	3
CaF_2	$\{001\}\ \langle 1\bar{1}0\rangle$	3
	$\{001\}\ \langle 1\bar{1}0\rangle + \{110\}\ \langle 1\bar{1}0\rangle$	5
TiO_2 (rutile)	$\{101\}\ \langle 10\bar{1}\rangle + \{110\}\ \langle 001\rangle$	4
$MgO \cdot Al_2O_3$, TiC	$\{111\}\langle 1\bar{1}0\rangle$	5

however, and as predicted this material lacks ductility in polycrystalline form.[14]

CaF_2 should behave in a manner similar to MgO. At low temperatures slip occurs on the {001} $\langle 1\bar{1}0 \rangle$ slip systems which provide three independent slip systems. At elevated temperatures slip also occurs on the {110} $\langle 1\bar{1}0 \rangle$ slip systems producing a total of five independent slip systems.[15] The mechanical behavior of single-crystal and polycrystalline CaF_2 is discussed by Pratt in his contribution to this volume. In TiO_2 (rutile), on the other hand, slip occurs on the {101} $\langle 10\bar{1} \rangle$ and the {110} $\langle 001 \rangle$ slip systems producing a total of four independent slip systems.[16] Thus, TiO_2 should be brittle in polycrystalline form. The {101} $\langle 10\bar{1} \rangle$ slip systems in TiO_2 are not equivalent to the {110} $\langle 1\bar{1}0 \rangle$ slip systems in NaCl because TiO_2 is tetragonal.

The family of slip systems for $MgAl_2O_4$ (spinel) and TiC is {111} $\langle 1\bar{1}0 \rangle$ which provides five independent slip systems; however, deformation occurs by slip only at elevated temperatures.[17,18] An interesting question with regard to both of these materials is the effect of stoichiometry on their flow stress. Both materials exist over a finite composition range, and thus it is possible that a large component of their flow stress is due to lattice defects resulting from nonstoichiometry. Recent results by Williams, however, show that in TiC the flow stress increases as the composition approaches that of the stoichiometric compound.[19]

III. GRAIN BOUNDARY SHEARING

At temperatures in the neighborhood of $T_{m/2}$, metals and probably most ceramics begin to exhibit some grain boundary sliding. Early investigations of MgO and Al_2O_3 by Wachtman and Lam and by Chang reported

changes in the elastic modulus.[20,21] Recently, more direct evidence of grain boundary sliding in ceramic materials was obtained by Adams and Murray who studied its occurrence in bicrystals of MgO.[22] They found that grain boundary sliding rates increased rapidly with increasing temperature and increasing stress, and that the resistance to grain boundary sliding depends on boundary orientation. In general, the more complex the boundary, the more readily sliding occurs.

A. Effect of Orientation and Temperature

Murray and Mountvala have made extensive measurements of the effect of temperature and orientation on grain boundary sliding in MgO.[23] Bicrystals were cut in the shape of rectangular parallelepipeds with the boundary lying at approximately 45° to the tensile axis. The boundary orientations were specified as illustrated in Fig. 5. The (1, 2, 3) and the (1', 2', 3') axes correspond to <100> directions in each crystal. The axis closest to the boundary normal is labeled 3. The axis closest to 3 is labeled 3'. The angle, α, between axis 3 and axis 3' is defined as the angle of tilt. To obtain β, the angle of twist, 3' is rotated to coincide with 3 so that (1, 2) and (1', 2') are coplanar. The angle between axis 1 and axis 1' is then defined as the angle of twist. It should be noted that this scheme gives atomic misorientations only for pure twist and pure tilt boundaries.

After determining their boundary orientations, the bicrystal specimens were loaded in compression and their fracture stress measured. In all cases the specimens were observed to fracture by shearing along the boundary.

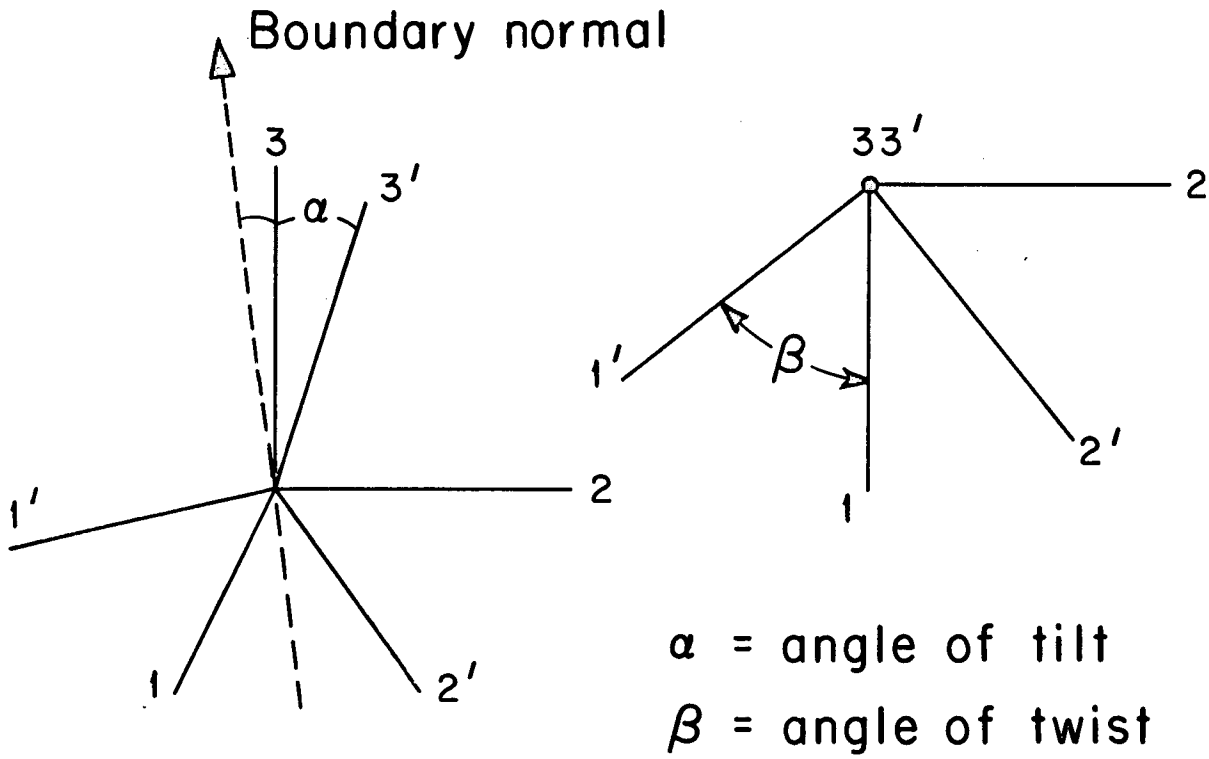

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Fig. 5. Scheme for describing grain boundary misorientation used by Murray and Mountvala.[23]

Figure 6 shows the effect of boundary orientation on fracture stress at 1400° C.[23] When the angle of tilt was less than 20°, the fracture stress decreased rapidly with increasing twist. When the angle of tilt was greater than 30°, the decrease was less pronounced.

Figure 7 shows the effect of temperature on fracture for several boundary orientations. It can be seen that the fracture stress decreases rapidly at a critical temperature which depends on boundary orientation. This critical temperature decreases with increasing twist and decreasing tilt.

From these results, it can be predicted that grain boundary sliding should start contributing to the deformation of polycrystalline MgO at about 1300° C. It should be emphasized, however, that such sliding at normal strain rates does not constitute a ductile mode of deformation because it is necessarily accompanied by the formation of accommodation cracks at three-grain junctions.

IV. DEFORMATION OF POLYCRYSTALLINE MgO*

Because of the extensive information available on mechanical behavior in single-crystal and bicrystal form, MgO was chosen for polycrystalline deformation studies.[22-27] MgO has the NaCl crystal structure. At low temperatures it is a material in which dislocations are mobile but which possesses an insufficient number of independent slip systems for ductility. At elevated temperatures MgO has five independent slip systems; however, ductility comparable to that observed in polycrystalline

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*This section, including the experimental results and discussion, is based on part of a paper by the authors on "Deformation of Polycrystalline MgO at Elevated Temperatures" which has been accepted for publication in the Journal of the American Ceramic Society.

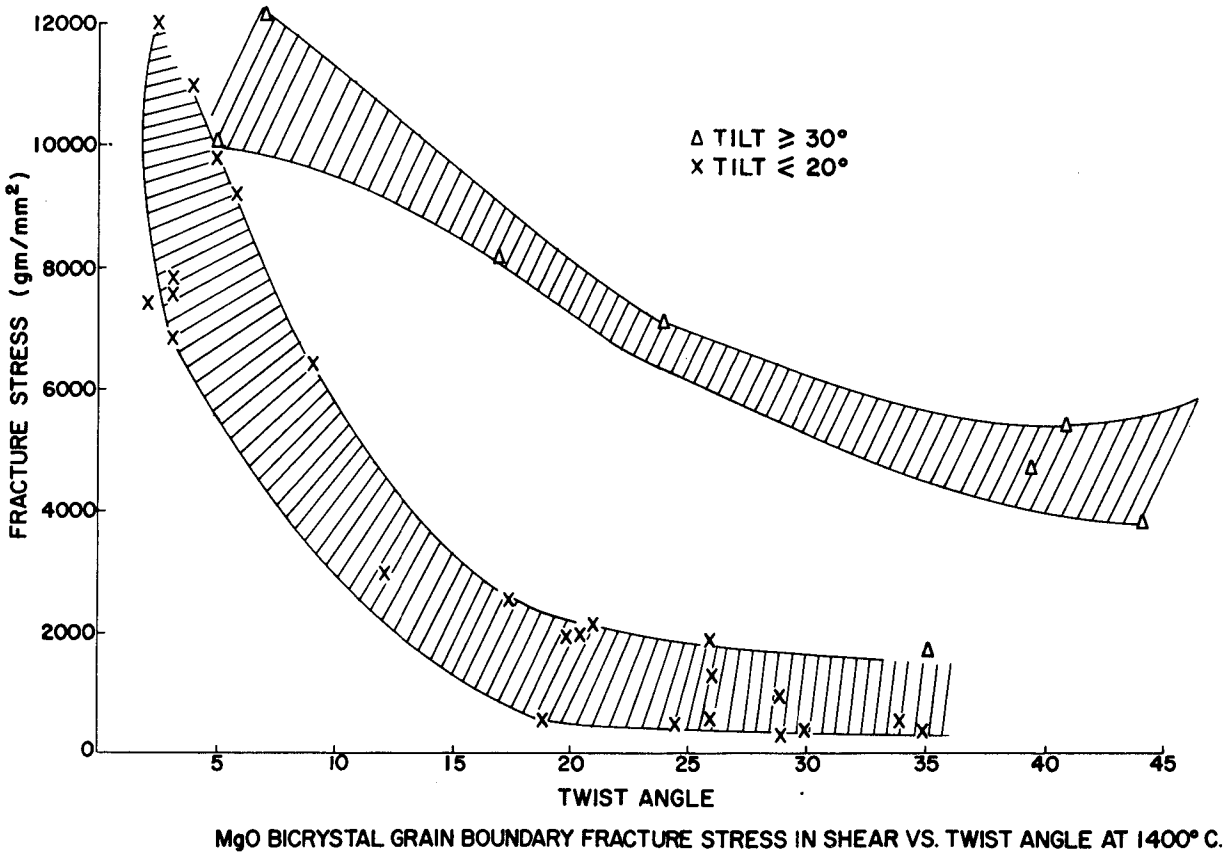
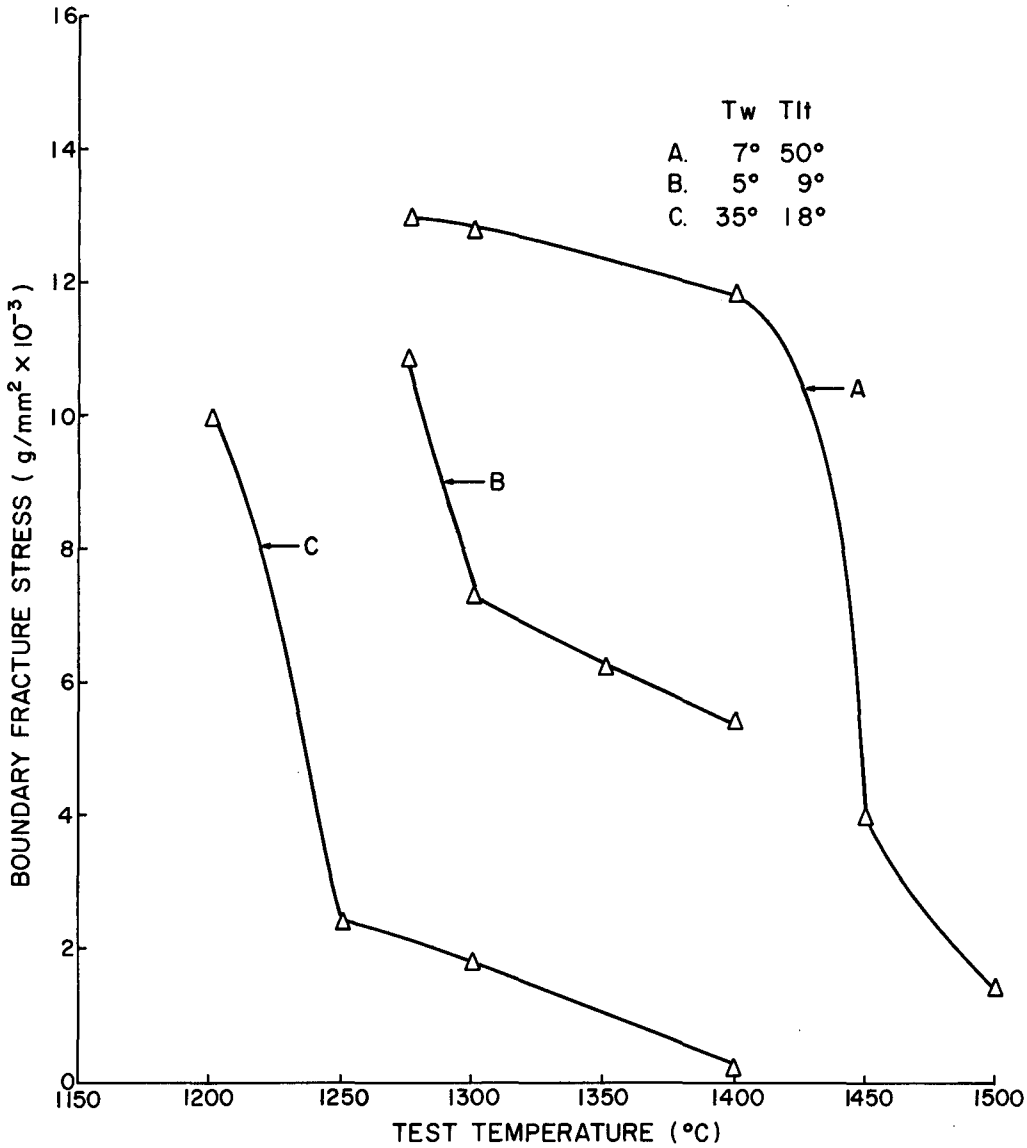


Fig. 6. The effect of grain boundary misorientation on strength (Murray and Mountvala[23]).



FRACTURE STRESS AS A FUNCTION OF TEMPERATURE FOR MgO BICRYSTALS

MUB-4353

Fig. 7. The effect of temperature on grain boundary strength (Murray and Mountvala[23]).

NaCl, AgCl, KCl, or LiF has not yet been reported.[12,25] On the basis of the observations of Murray and Mountvala, grain boundary sliding should appear in polycrystalline MgO above 1300° C. In this section, the results of stress-strain experiments on two different types of polycrystalline MgO are presented. Both types were nominally dense and pure but they deformed differently, thus emphasizing the importance of subtle differences in structure* in determining mechanical behavior.

A. Experimental Procedure

1. Specimens

The composition of each type of polycrystalline MgO was determined by spectrographic analysis. The constituents are reported in Table II as oxides of the elements.

Type 1 was made in the authors' laboratory using a technique based on one described by Rice.[28] In brief, a mixture of powdered MgO with 3% LiF was hot-pressed at 1000° C at a pressure of 3000 psi for about 30 min in a graphite die in a vacuum (10^{-3} mm Hg). The resulting compact was then fired at 1300° C for 4 hrs in air. This procedure resulted in a transparent polycrystalline MgO containing approximately 75 ppm of Li† with a density of 3.579 g cm^{-3} § and with the microstructure shown in Fig. 8. The grain size was uniform, the largest cross sections being

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*Structure is used in the broad sense to include composition, microstructure, flaws, etc. Microstructure refers to the geometric distribution of identifiable grains and phases.

†Private communication from Roy W. Rice states that radio-tracer techniques indicate that such specimens contain approximately 0.08% F.

§All reported density values were obtained by use of Archimedes principle and are thus comparable; x-ray density value for MgO single crystals is 3.5733.

Table II. Spectroscopic analysis of polycrystalline MgO*

Type	Mg	Fe	Ba	Si	Mn	Al	Ca	Cu	Ti	Li	Ni	Cr	B
1	Principle	--	--	0.01%	--	< 0.005	0.003	0.0008	0.006	0.0075	--	0.004	--
2	Principle	--	--	0.015%	--	< 0.005	0.02	0.0008	--	--	--	--	--

*Analyses made by the American Spectroscopic Laboratories, San Francisco, California. Constituents reported as oxides of the elements indicated.

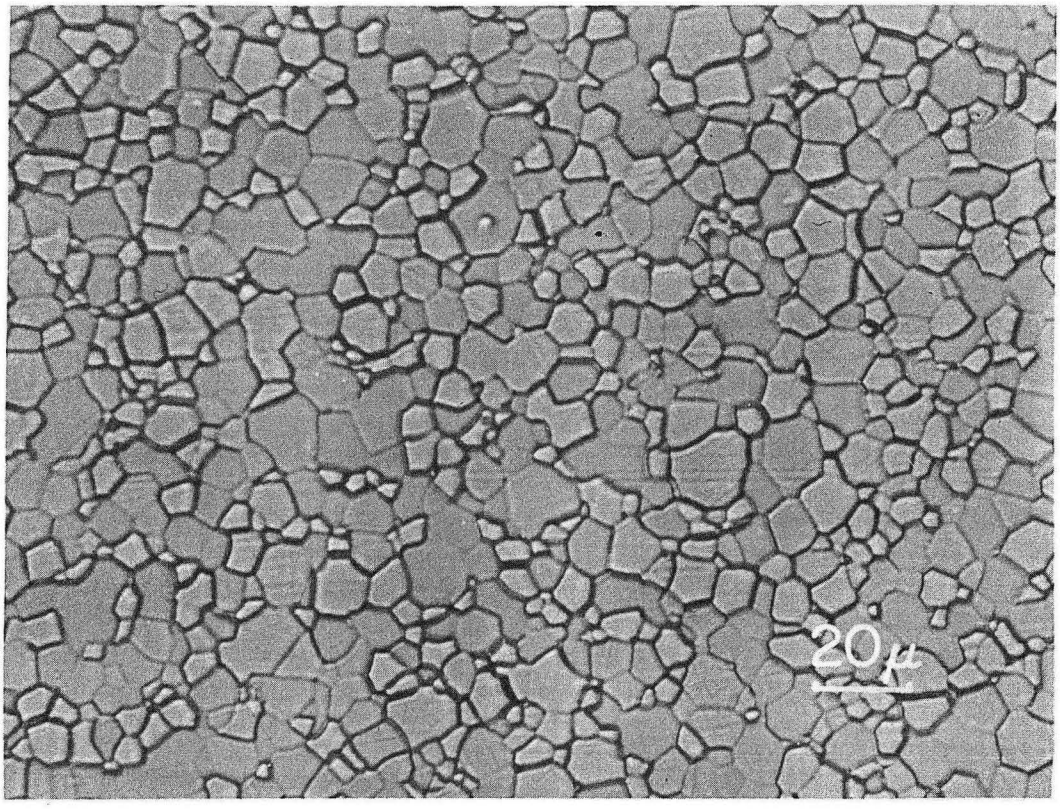

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Fig. 8. Structure of Type 1 polycrystalline MgO.

about 15μ. These specimens were called Type 1 (S.G.). Some of this type was given a high temperature anneal to cause grain growth. After this anneal the grains varied in size from region to region, and grain cross sections up to 100μ were observed. These specimens were called Type 1 (L.G.).

Type 2 was obtained from the Honeywell Research Center, Hopkins, Minnesota, and was formed by an isostatic pressing and sintering method. It was white and translucent with a density of 3.546 and with the microstructure shown in Fig. 9. A number of very small pores can be seen within the grains and on the grain boundaries. The largest grain cross sections are about 50μ.

Compression specimens of each type of polycrystalline MgO were cut with a diamond saw. The specimens were prisms with approximately square cross sections about 0.2 by 0.2 by 1.0 in. Coarse abrasive paper (220A) was used to grind specimen ends parallel.

2. Stress-Strain Experiments

Stress-strain curves were obtained for each type of MgO at a number of constant temperatures. Specimens were loaded in compression at a constant force rate such that the initial stress rate was 20 psi/sec. The stresses were calculated from initial specimen cross sections. Strains were determined by measuring the displacement of two small divot holes initially 0.5 in. apart on a side face of the specimen. All reported strains are true strains, the true strain being equal to the natural logarithm of one plus the engineering strain.

Specimens were heated in air in a furnace with $MoSi_2$ heating elements. Alumina buttons were placed between specimen ends and the loading rams, which were also made of alumina. Thin platinum sheets were placed

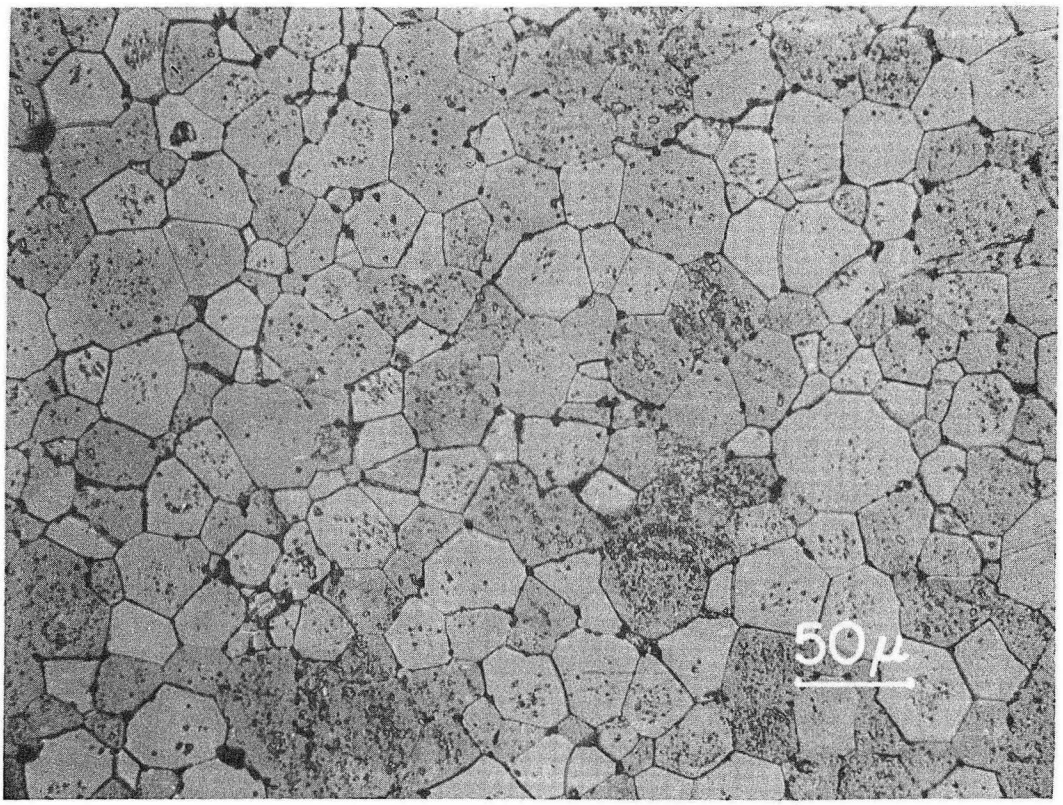

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Fig. 9. Structure of Type 2 polycrystalline MgO.

between the specimen ends and the buttons as reaction barriers. A detailed description of the overall apparatus used in this investigation will be published separately.[29]

3. Examination of Specimens

Several specimens were carefully polished on one face to allow microscopic examination before and after deformation. The faces were (a) flattened by grinding with 600-grit SiC, (b) lapped with 1-2μ diamond grit, and (c) lapped with a high speed wheel using Linde A on Politex Polishing Disks (Style PA-K).* All specimens were chemically polished for 1 min in 85% orthophosphoric acid and 1 part sulfuric acid. MgO single-crystal specimens immersed in this etch for 1 min at 26°C showed pits only on the {100} and {110} faces.

All photographs were taken with a Leitz Metallographic Microscope (MM5) using 4 by 5 in. Polaroid 55P/N film.

B. Results

In a force rate experiment, the slope of the stress-strain curve is related to the strain rate at small strains by the equation

$$\frac{d\sigma}{d\epsilon} = \frac{d\sigma}{dt}\left(\frac{d\epsilon}{dt}\right)^{-1}, \qquad (6)$$

where σ is the applied force divided by the cross section of the undeformed specimen, ϵ is the strain, and t is time. It can be seen that for a constant force rate a small slope of the curve indicates a high strain rate; and a high slope, a low strain rate.

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*Geoscience Instrument Corp.

1. Type 1 Specimens

Figure 10 shows typical stress-strain curves for Type 1 (S.G.) specimens at temperatures from 1000 to 1500°C. At 1000°C fracture occurred with little bulk strain, and the basic shape of the curve is different from those obtained at higher temperatures. At 1400 and 1500°C, after initial yielding, the stress-strain curves rapidly became flat indicating high strain rates; and the experiments were terminated by fracture of the alumina supporting buttons. At 1200 and 1300°C stress-strain curves are intermediate and terminated by fracture of the specimen.

After deformation it was noticed that the Type 1 (S.G.) specimens had lost their transparency, the 1400 and 1500°C specimens being appreciably opaque. These observations suggested that deformation might be occurring by grain boundary shearing and separation. To check this hypothesis, lines approximately 1μ wide were scribed with a diamond point on the mechanically and chemically polished surface of a Type 1 (S.G.) specimen. Figure 11 shows the surface of this specimen after it was strained 3% at 1400°C. The displacement of the scribed lines between grains A and B is clear evidence of grain boundary shearing. Of particular interest is the cleavage fracture developing in grain A. Such fracture is a necessary consequence of grain boundary shearing and allows for accommodation at three-grain junctions. Although occasionally occurring within grains (as in grain A), such localized fracture occurred most frequently along the grain boundaries.

A second feature of interest in Fig. 11 is the ghost lines which were not present before the deformation experiment. These lines are the result of thermal etching and correspond to the positions where the

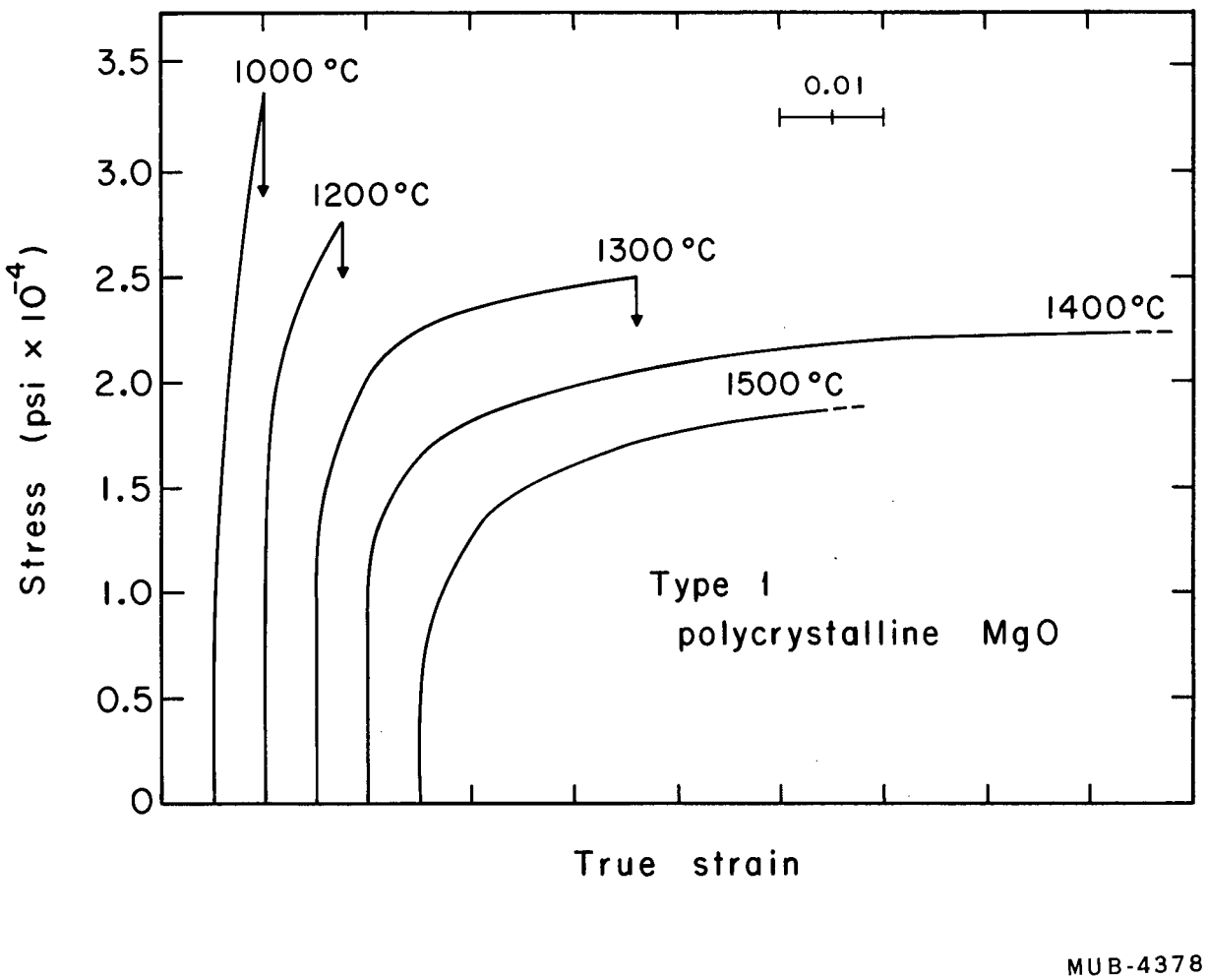
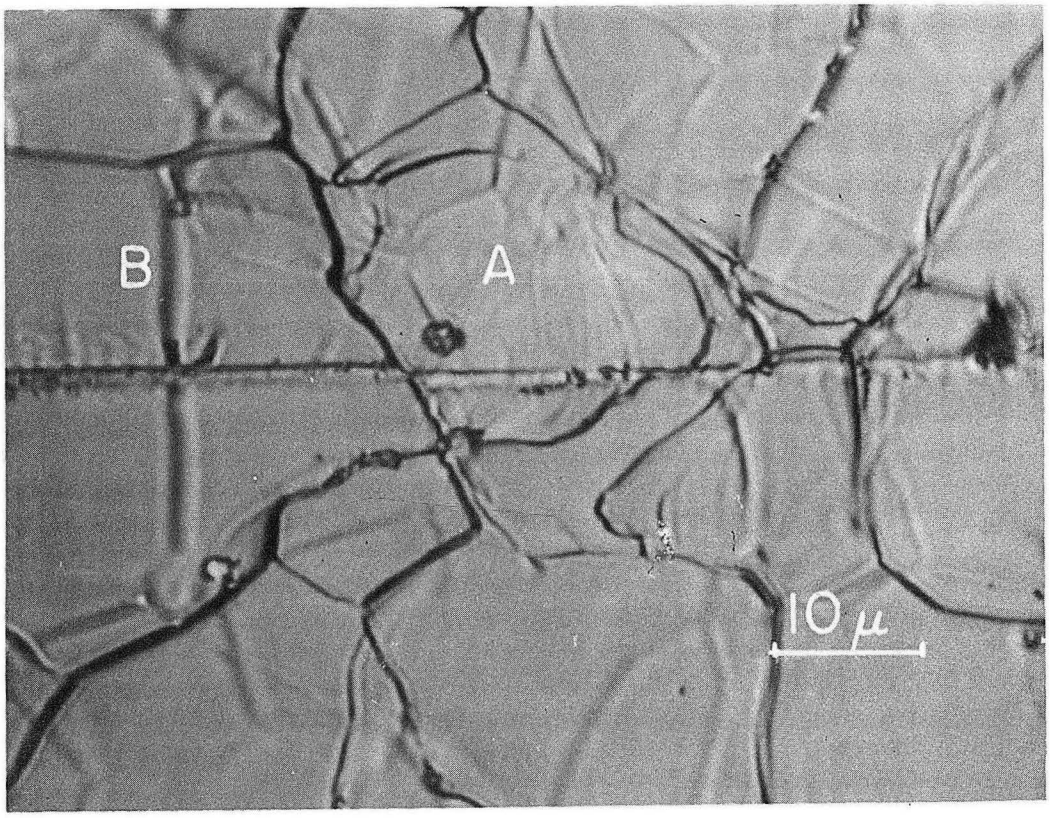


Fig. 10. Stress-strain curves for Type 1 specimens at temperatures ranging from 1000 to 1500° C.



ZN-4707

Fig. 11. The displacement of a scribed line indicating grain boundary sliding in a Type 1 specimen.

grain boundaries were stationary for a period of time at 1400° C. The final grain boundary positions are darker than the ghost lines. They are sharp bottomed due to the presence of the boundary, while the ghost lines are rounded due to additional thermal polishing after the boundary moved.

The translucency of the deformed Type 1 (S.G.) specimens is thus accounted for by localized fracture and grain boundary separations. These discontinuities also account for the high strain rates shortly after yielding because they reduce the cross-sectional area resisting deformation. This effect is emphasized with increasing strain because as the amount of localized fracture increases, the gaps between grains join together to form long intergranular cracks roughly parallel to the loading axis.

Figure 12 shows an intergranular crack in a Type 1 (L.G.) specimen deformed to fracture at 1300° C. Although little bulk straining by slip actually occurred, dislocation etch pits can be seen on the favorably oriented grains. In a large grain specimen, the gaps resulting from grain boundary shearing are quite large and can spread easily causing bulk fracture. Thus, from 1000 to 1500° C, the Type 1 (L.G.) specimens were observed to fracture with little plastic strain.

Figure 13 shows cylindrical voids which were frequently observed on grain boundaries which had slid. These voids probably mark the position of steps in the boundary which may have been produced by slip. The cylindrical shape of these voids can be seen because grains of different orientation were attacked by the polish at different rates, thus revealing the boundaries of the slowly attacked grains.

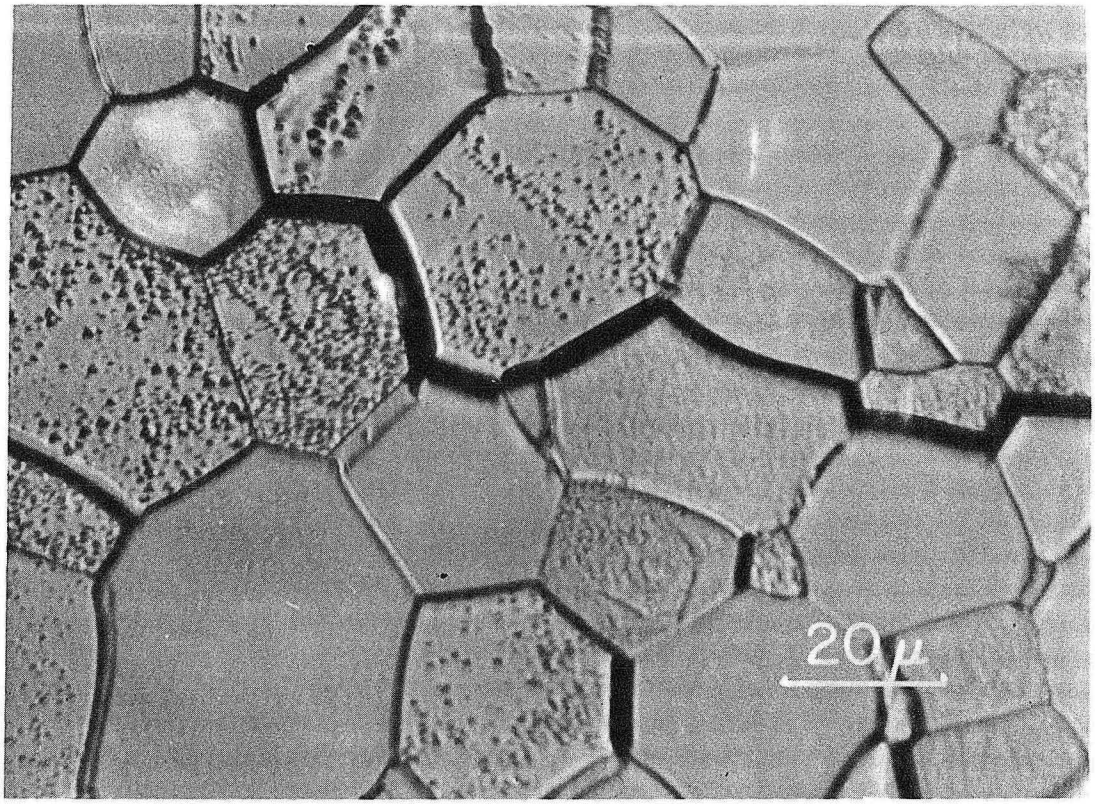


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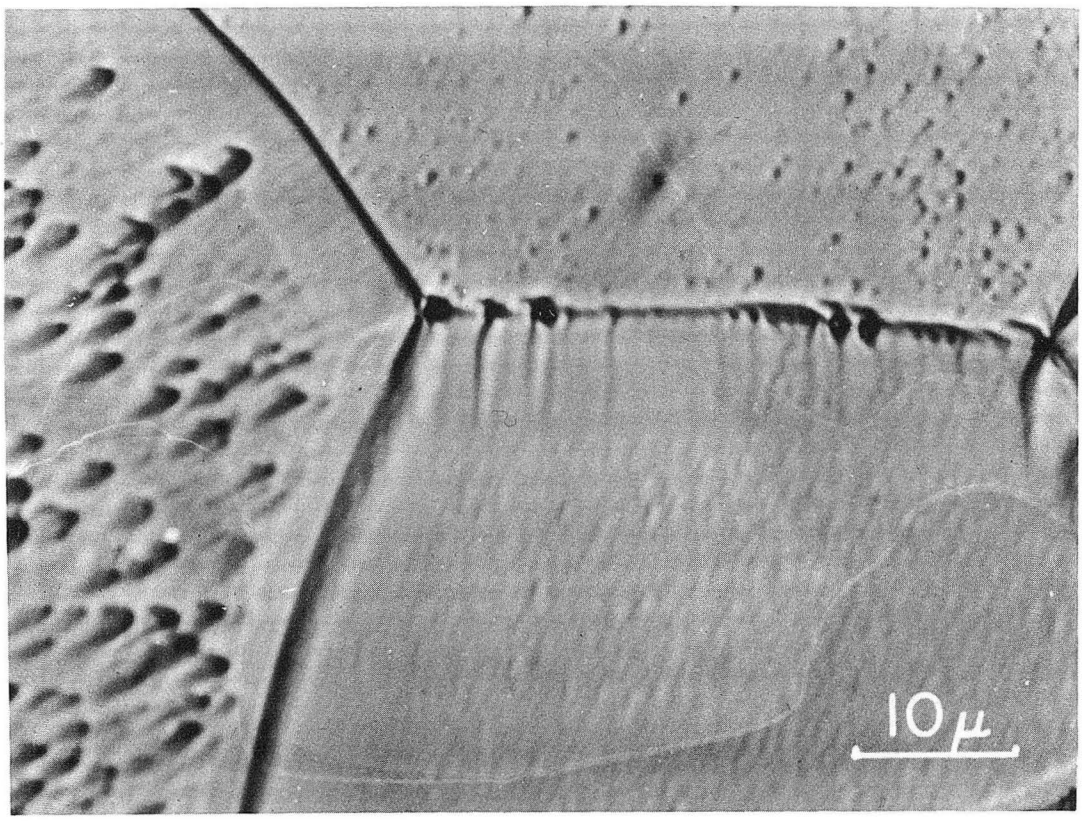
Fig. 12. Intergranular cracking in a Type 1 specimen deformed at 1300° C.



ZN-5002

Fig. 13. Cylindrical voids marking boundaries where sliding has occurred in a Type 1 specimen.

2. Type 2 Specimens

Figure 14 shows typical stress-strain curves for the Type 2 specimens at temperatures from 400 to 1400° C. Experiments were stopped prior to fracture to obtain complete specimens for microscopic examination. It can be seen that the Type 2 specimens were ductile well below 1200° C. Between 800 and 1200° C, parabolic-shaped stress-strain curves were obtained. At 1400° C, however, the curve was similar to that for the Type 1 (S.G.) specimen in that it rapidly became flat after initial yielding, suggesting that grain boundary shearing was occurring at this temperature. At 400° C the stress-strain curve was not parabolic and little bulk strain occurred, while at 600° C it was intermediate in nature. Figure 15 shows the surface of a specimen deformed at 400° C. Many grain boundaries were cracked; however, little intragranular cracking was observed.

Figure 16 shows the surface of a Type 2 specimen deformed about 5% at 1000° C. This surface had been mechanically and chemically polished prior to deformation but was not treated after deformation. The dark spots were caused by chemical polish and were visible on the surface before deformation. The wavy lines appeared after deformation and are similar to the "wavy slip" steps observed previously in NaCl, AgCl, KCl, and LiF. These steps were quite pronounced and appeared in almost all of the grains. They occasionally crossed grain boundaries and often became forked as they approached grain boundaries. Similar steps were observed on specimens deformed at 800° C but not at 600° C. The 1200 and 1400° C specimens were not prepared for this observation.

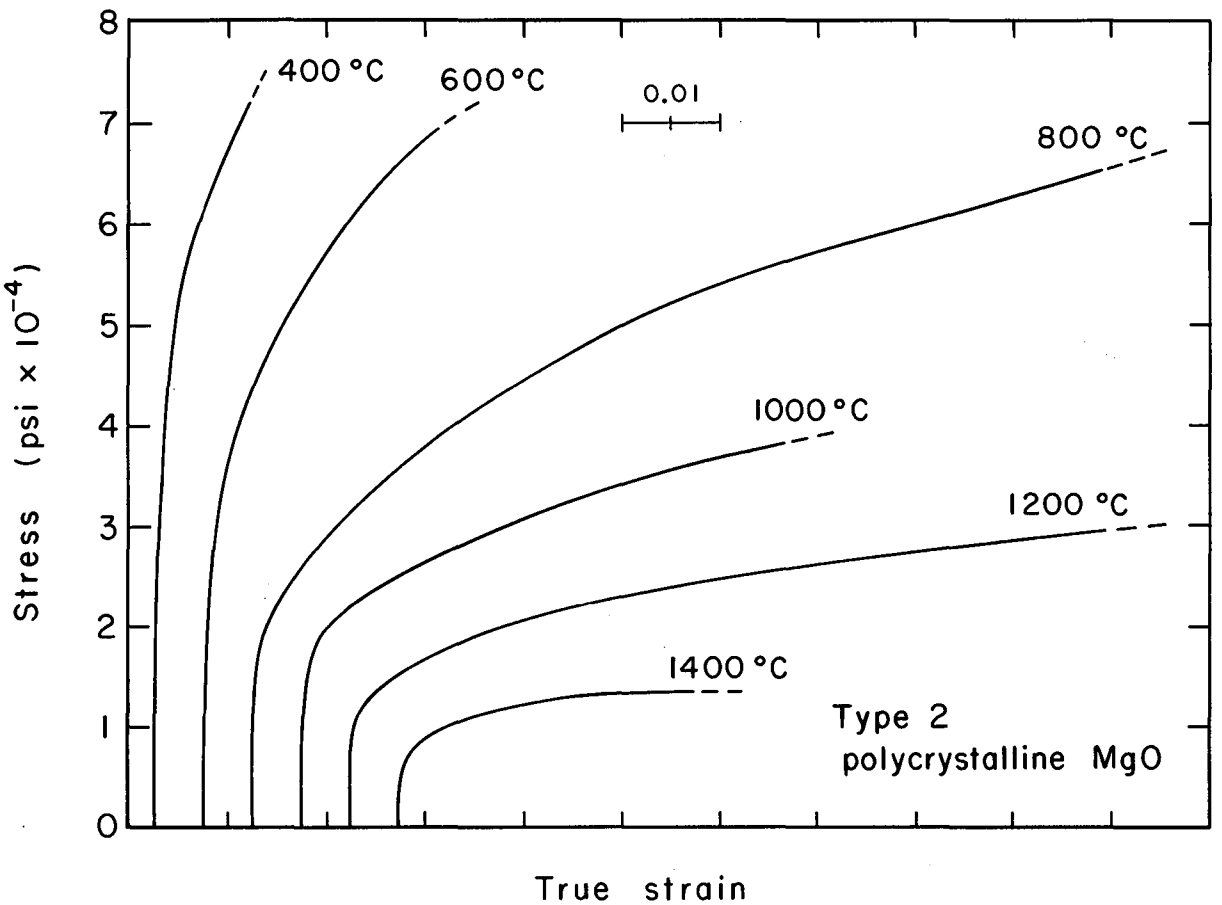


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Fig. 14. Stress-strain curves for Type 2 specimens at temperatures from 400 to 1400° C.

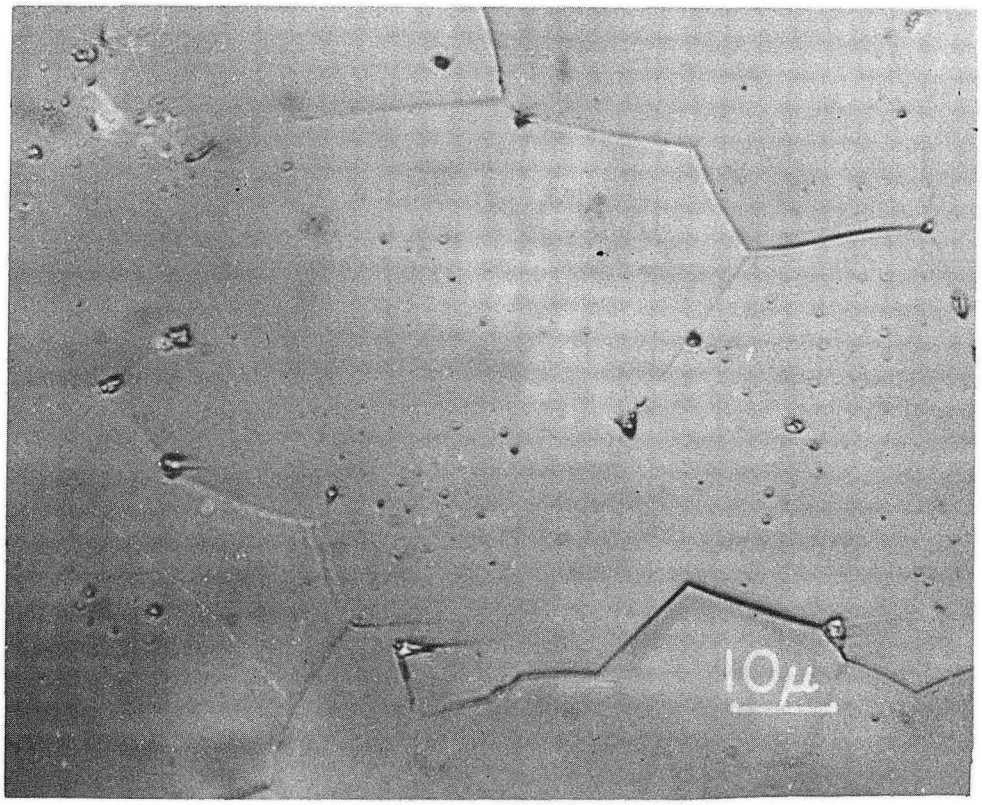

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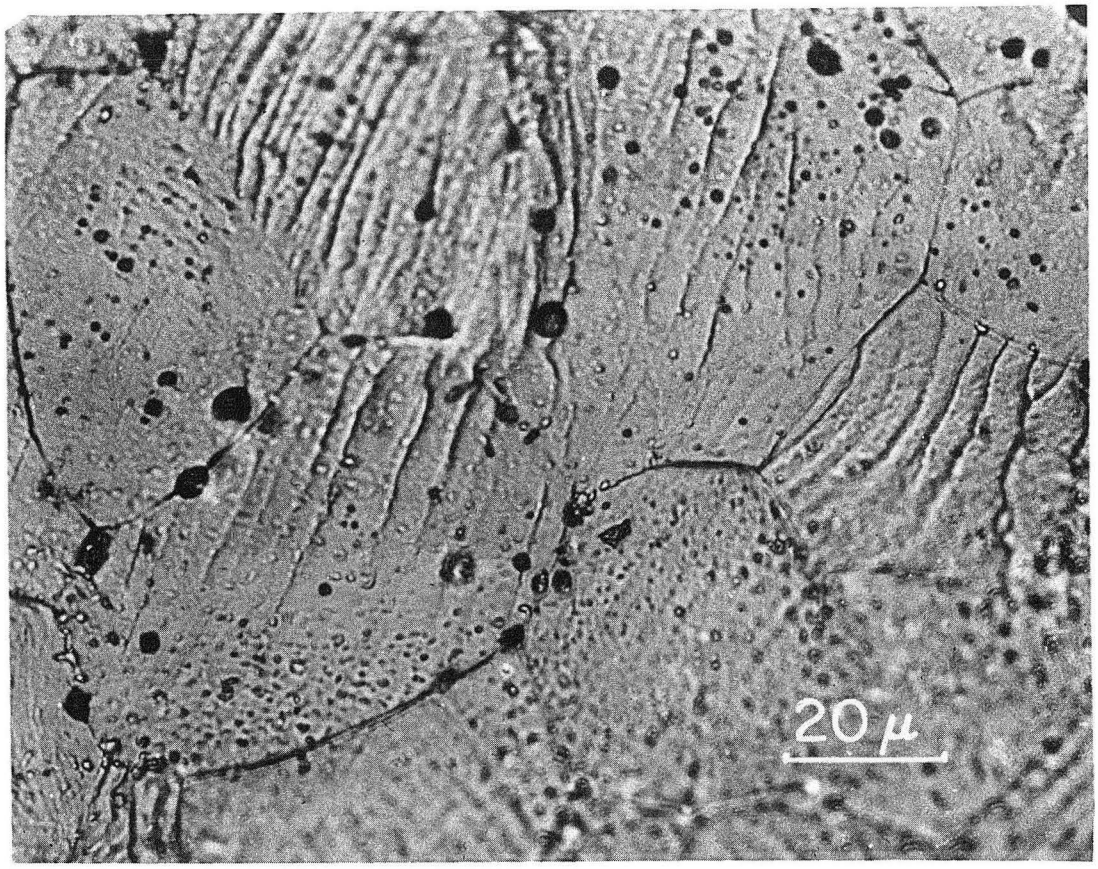
Fig. 15. Structure of a Type 2 specimen deformed at 400° C.



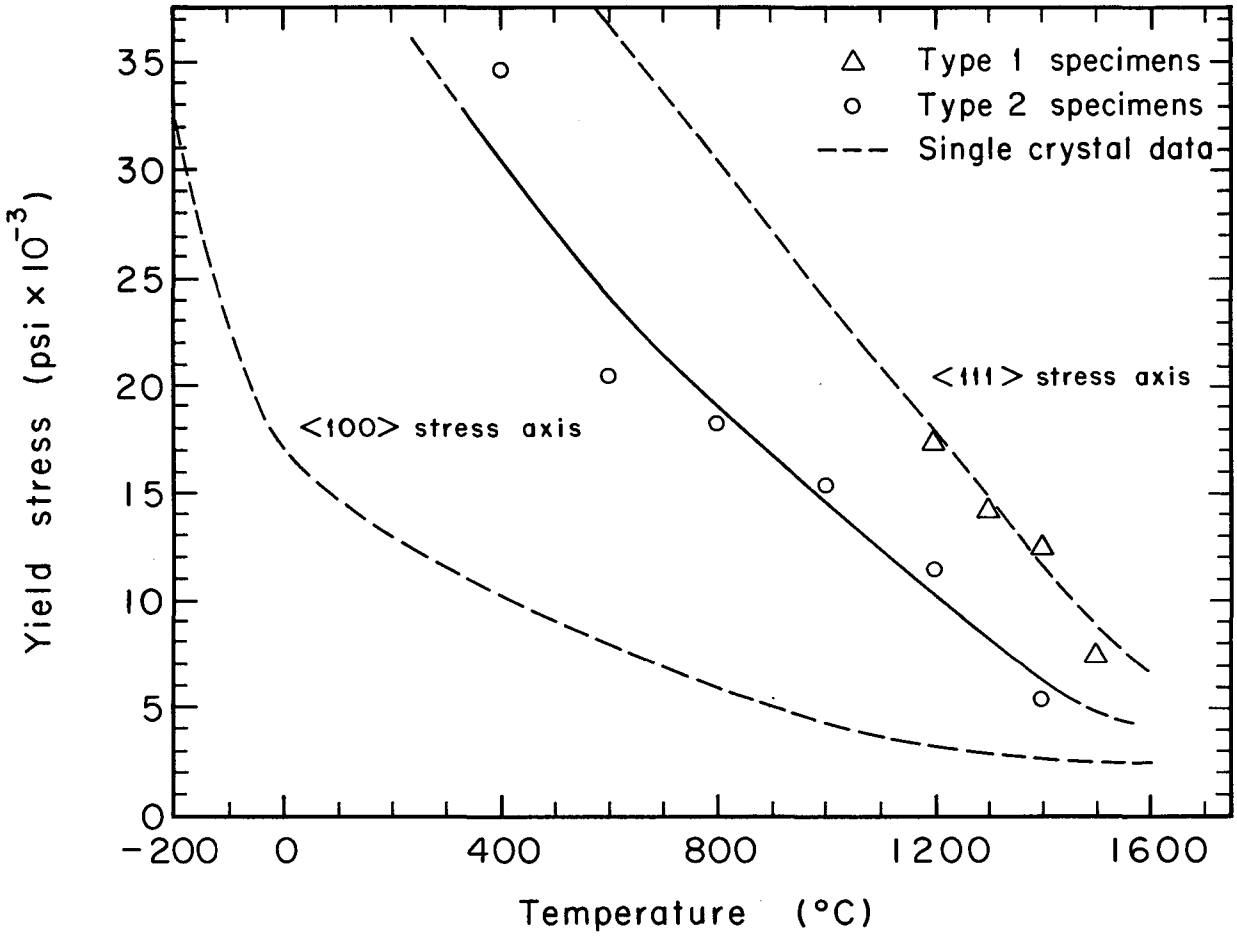
ZN-4713

Fig. 16. "Wavy slip" in a Type 2 specimen deformed at 1000° C.

C. Discussion

Of the two types of specimens studied in this investigation, only the Type 2 exhibited behavior comparable to that previously reported for AgCl, NaCl, KCl, and LiF. At 600° C and below these specimens were brittle and their deformation was accompanied by a pulling apart of grain boundaries. At 800° C and above they were ductile and wavy slip steps were observed. A transition from brittle to ductile behavior thus occurred between 0.28 T_m and 0.35 T_m. In AgCl, NaCl, and KCl similar transitions were observed in tension at about 0.24 T_m, 0.44 T_m, and 0.40 T_m, respectively.[6-8] In LiF a similar transition was observed in compression at about 0.67 T_m.[9]

Before comparing the behavior of the two types of polycrystalline MgO, the behavior of the Type 2 specimens will be considered in more detail. The deformation of these specimens can be further understood by comparing their stress-strain behavior to that of uniaxially stressed single crystals. Figure 17 gives yield stress values for MgO single crystals with <100> and <111> stress axes as a function of temperature.[25,26] A <100> stress axis results in equal, nonzero resolved shear stresses on four {110} $\langle 1\bar{1}0 \rangle$ slip systems. A <111> stress axis results in equal, nonzero resolved shear stresses on three {001} $\langle 1\bar{1}0 \rangle$ slip systems. In both orientations the resolved shear stress on all other {110} $\langle 1\bar{1}0 \rangle$ and {001} $\langle 1\bar{1}0 \rangle$ slip systems is zero. The ratio of yield stresses for single crystals with a <111> stress axis to those with a <100> stress axis provides a measure of the relative mobility of dislocations on the two families of slip systems. This ratio is 13:1 at 350° C, 4.5:1 at 600° C, and 2.9:1 at 1600° C. Because slip on both families of slip systems is



MUB-4377

Fig. 17. Yield stress vs. temperature curves for ⟨100⟩ and ⟨111⟩ oriented single crystals and for the Types 1 and 2 polycrystalline specimens.

necessary to satisfy the von Mises requirement, an increase in ductility with increasing temperature would be predicted.

Also shown in Fig. 17 are the yield stress values for the Types 1 (S.G.) and 2 polycrystalline specimens. It can be seen that the Type 2 specimens yielded at stresses considerably below those necessary to yield a single crystal with a <111> stress axis. This fact is particularly significant because a <111> stress axis produces a near maximum resolved shear stress of 0.472 times the uniaxial stress on the three {001} $\langle 1\bar{1}0 \rangle$ slip systems. Thus, slip on these slip systems in the randomly oriented grains of the Type 2 specimens could not have resulted from the uniaxial stress alone but is evidence of additional stresses due to interactions between grains.

These observations suggest the following explanation for the yielding behavior of the Type 2 specimens. With the application of stress, slip occurs first on the {110} $\langle 1\bar{1}0 \rangle$ slip systems because of their lower resistance to dislocation motion. Slip on these slip systems can provide, however, only two independent slip systems so that long range stresses build up in each grain. Above the brittle-ductile transition temperature these stresses force slip on the {001} $\langle 1\bar{1}0 \rangle$ slip systems and yielding occurs in the polycrystalline piece. (This phenomenon was quantitatively demonstrated for large grain polycrystalline LiF by Scott and Pask wherein a localized shear stress of 9000 psi was shown to develop on a slip system on which the resolved shear stress due to the applied load was only 880 psi.[30]) Below the brittle-ductile transition temperature these stresses cause grain boundary separation before they become sufficient to cause slip on the {001} $\langle 1\bar{1}0 \rangle$ slip systems and thus the specimen fractures without yielding. It follows that the brittle-ductile transition

temperature in polycrystalline MgO should depend not only on the mobilities of dislocations on the {110} <1$\bar{1}$0> and the {001} <1$\bar{1}$0> slip systems but also on the strength of the grain boundaries.

It is also interesting to compare the strain hardening behavior of the Type 2 polycrystalline specimens to that of uniaxially stressed single crystals in the temperature range where polycrystalline deformation occurred predominantly by slip (800-1200°C). According to earlier measurements by Copley and Pask, single crystals compressed with <100> stress axes exhibit much less strain hardening than the Type 2 specimens.[26] Although four slip systems are equally stressed in this orientation, slip in a particular volume element of the crystal will generally occur on only two slip systems with orthogonal Burgers vectors. The interpenetration of slip on slip systems with Burgers vectors enclosing an angle of 60° (or 120°) is observed only where a transition occurs from one set of orthogonal slip systems to the other. Long range stress interactions exist between dislocations on these slip systems, and dislocations with Burgers vectors at 120° can react to form sessile dislocation networks which are observed where such transitions occur.[26,27,31] These observations suggest that greater strain hardening should result from interpenetrating slip on the oblique slip systems than on the orthogonal ones.

Figure 18 shows stress-strain curves obtained by compressing single crystals of MgO with <100>, <111>, and <110> stress axes at 1300°C. In the <111> orientation, slip occurs on three {001} <1$\bar{1}$0> slip systems providing that the ends are constrained from lateral movement. The Burgers vector of each of these slip systems is at 60° (or 120°) relative to the other two. In the <110> orientation slip occurs on four

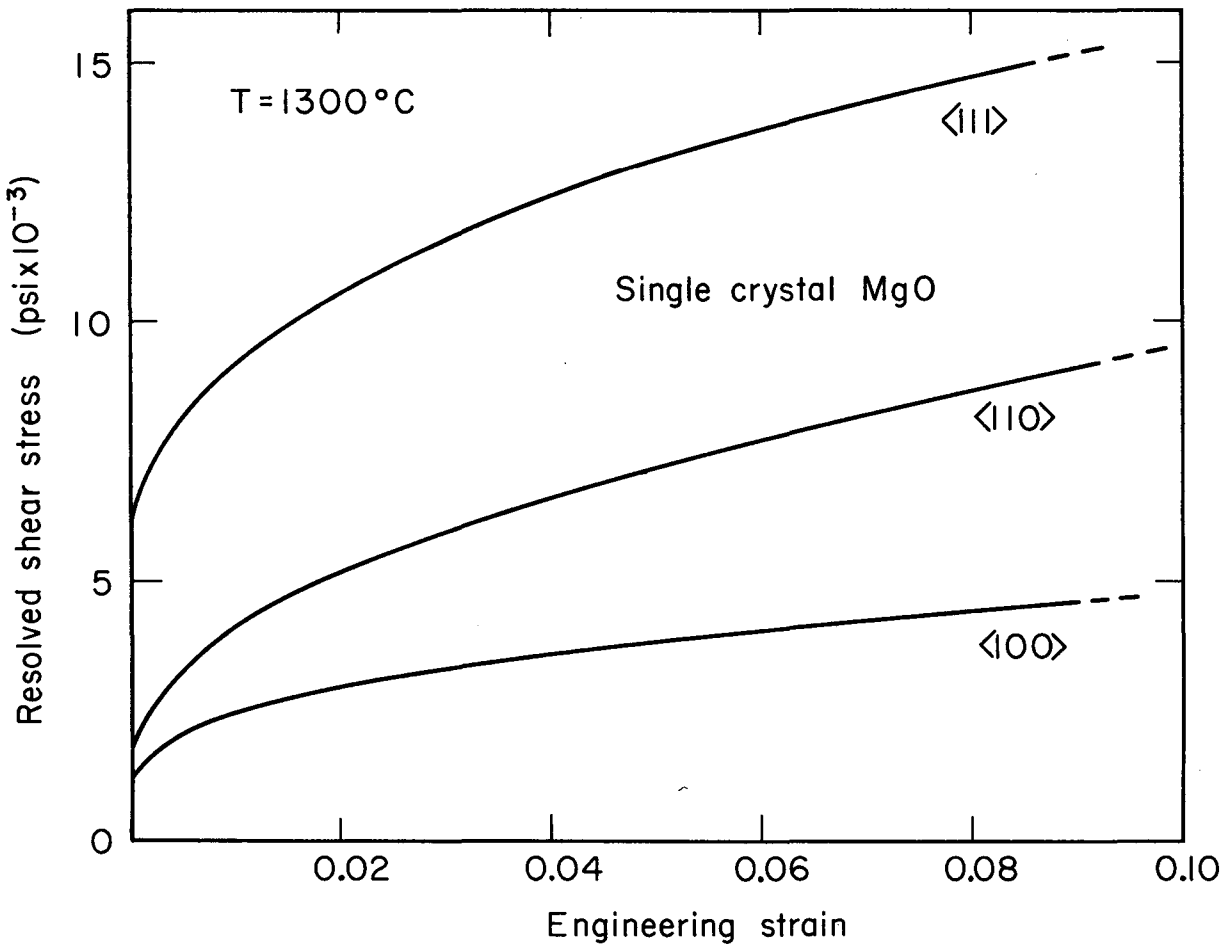


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Fig. 18. Resolved shear stress-strain curves for single crystals with ⟨100⟩, ⟨110⟩, and ⟨111⟩ stress axes at 1300° C.

{110} <1$\bar{1}$0> slip systems. The interpenetration of slip on slip systems with Burgers vectors enclosing an angle of 60° (or 120°) is again necessary if the specimen ends are constrained. It can be seen that the critical resolved shear stress is greater for the {001} <1$\bar{1}$0> than for the {110} <1$\bar{1}$0> slip systems at 1300°C and that strain hardening is greater when the interpenetration of 60° (or 120°) slip is required.

In polycrystalline MgO, slip on five independent slip systems in each grain is necessary for ductility and thus an interpenetration of slip on the oblique slip systems must occur. A measure of the amount of strain hardening that might be expected under these conditions is provided by the stress-strain behavior of single crystals with <111> and <110> stress axes. A comparison of the stress-strain curves for the <111> and <110> oriented single crystals shown in Fig. 18 to those for the Type 2 specimens shows that the strain hardening in all three cases is comparable.

After considering in some detail the Type 2 polycrystalline specimens, their behavior will now be compared to that of the Type 1 (S.G.) specimens. Both specimen types were relatively pure and lacked a detectable second phase. They differed, however, in grain size, in the presence of Li and F in the Type 1 specimens, and in the presence of a dispersion of fine pores in the Type 2 specimens. The Type 1 specimens fractured without significant deformation at 1000°C and below, while the Type 2 specimens exhibited ductile behavior down to 800°C. Yielding was observed in the Type 1 (S.G.) specimens only at 1200°C and above where they deformed primarily by grain boundary shearing. As shown in Fig. 17, the Type 1 (S.G.) specimens generally supported greater stresses than those required to yield the Type 2 specimens without yielding themselves.

The less-ductile behavior of the Type 1 specimens may be caused by (a) lower dislocation mobility, (b) greater difficulties in initiating slip, or (c) grain boundary weakness. A reduction of the mobility of dislocations may be caused by the presence of Li and F. Gorum, Luhman, and Pask have shown that small solute additions can increase the yield stress of MgO single crystals by as much as four times.[32] Microhardness measurements on both types of specimens, however, detected no difference in hardness.

If dislocations and slip are initiated at only a few points in a grain, they will cause nonhomogeneous stress concentrations and fracture. Stokes and Li on the basis of room temperature tensile experiments on polycrystalline MgO concluded that mobile dislocations can be introduced through stress concentrations associated with pores.[33] Thus, it is possible that the presence of the small amount of pores in the Type 2 specimens may play a significant role in initiating slip.

Lastly, the presence of Li and F in the Type 1 specimens, preferably at grain boundaries, can be expected to have a significant effect on the structure, strength, and behavior of the grain boundaries. Modifications in their structure can increase the difficulties in forcing slip on $\{001\}\langle 1\bar{1}0\rangle$ slip systems in adjoining grains. On this basis, the Type 2 specimens have grain boundaries whose strength and characteristics are considerably more favorable for allowing the forcing of slip in $\{001\}\langle 1\bar{1}0\rangle$ slip systems and the development of wavy slip. The greater ductility of the Type 2 specimens than Type 1 (S.G.) does not appear to be attributable to some unknown favorable role of their larger grain size since the Type 1 (L.G.) specimen, whose grain size is still larger, showed no ductility at 1000° C.

V. DUCTILITY IN POLYCRYSTALLINE CERAMICS

A. Crack Formation

The demonstration by Gorum, Parker, and Pask that MgO single crystals exhibited substantial ductility at room temperature stimulated considerable interest in developing a ductile, refractory, polycrystalline ceramic material.[34] Initial studies were directed toward elucidating the process of crack nucleation in single crystals of MgO. Stokes, Johnston, and Li showed that cracks form at slip band intersections parallel to {110} planes in both compression and bend specimens.[35-37] Once formed, the behavior of these cracks was found to depend on the distribution of slip in the crystal.[38] If slip was confined to only a few slip bands, the cracks were found to spread rapidly causing fracture. If many slip bands were present, fewer cracks were nucleated and the growth of these cracks was frequently observed to cease when they crossed neighboring slip bands even though their length exceeded that necessary for propagation as a Griffith's crack. Such stabilization was attributed to the additional energy necessary to propagate a crack across a slip band. As pointed out by Gilman, the surface area of a crack is increased in such a region due to formation of steps where screw dislocations are cut and thus more strain energy must be expended.[39]

The nucleation of cleavage cracks in MgO was at first believed to result from the coalescence of edge dislocations piling up at a barrier, a mechanism first proposed by Zener[40] and analyzed mathematically by Stroh.[41,42] It soon became apparent, however, that this mechanism alone could not completely explain the observed behavior. It could not explain, for example, why cracks were formed only at intersections where complete interpenetration of slip bands occurred. Recently, many such details of

crack formation in MgO single crystals have been clarified by Argon and Orowan.[43] They show that high stresses of a macroscopic nature exist where dislocation bands intersect and that these stresses, although often reduced by plastic deformation in the form of kinking, play a dominant role in the formation of the observed cracks.

Since cleavage cracks were observed to form at slip band intersections, it was expected that they would also form in polycrystalline MgO where slip bands would almost certainly be blocked by grain boundaries. This behavior was confirmed in experiments on MgO bicrystals by Johnston, Stokes, and Li[44] and by Westwood.[45] Johnston et al. showed, in fact, that a direct correlation existed between the fracture stress of MgO bicrystals and the stress required to form slip bands in them. Ku and Johnston[46] (see also Johnston and Parker[47]) have recently studied the stress necessary to form a crack at the intersection of a slip band and a grain boundary in MgO in greater detail. Slip bands were initiated at microhardness indentations on the surfaces of bicrystals which had been extensively polished to remove all other dislocation sources. It was found that the fracture stress of the bicrystals σ_f obeyed the well-known Petch equation

$$\sigma_f = \sigma_o + A\, L^{-1/2} , \qquad (7)$$

where in this case L was equal to the distance from the microhardness indentation to the boundary. It was also found that a physical significance could be attached to σ_o and A. The constant σ_o was found for all boundaries studied to equal the stress required to form a slip band. The constant A could be correlated to the strength of the grain boundary. This result suggests that crack nucleation might be suppressed in

polycrystalline MgO by reducing the grain size as proposed by Parker several years ago, and that the effect of reducing the grain size will be greatest in the material with the strongest grain boundaries.[48]

B. Dislocation Sources and Distribution of Slip

Although much progress has been made in understanding the relationship of slip to crack formation in single crystals and bicrystals of MgO, several important facts remain to be established in the case of polycrystalline MgO. One of the more important of these is the distribution of slip in the grains of polycrystalline MgO deformed at various temperatures. If slip occurs inhomogeneously, there is little chance of ductility. If it occurs homogeneously, however, the probability of nucleating cracks is reduced and the probability of stabilizing cracks, already formed, is greater.

The distribution of slip depends on the number of sources (i.e., segments of mobile dislocation line) present in each grain. The number of such sources present in a single-phase ceramic material should ordinarily be small. Although they are often deformed at some stage of their preparation (hot-pressing of the Type 1 specimens described in Section IV-A-1), ceramic materials are generally annealed at high temperatures to obtain maximum density. The grain growth resulting from this anneal generally removes all dislocation substructure from within the grains. Thus, the distribution of slip in such materials should depend on the capacity of the grain boundaries to act as sources of dislocations.

Although grain boundaries in metals are known to act as dislocation sources, there is no direct evidence that they act as sources in ceramic materials. In metals, the energy of a boundary joining grains with a

substantial mismatch should vary only slightly with the change in orientation accompanying the emission of a dislocation. On the other hand, the same change in orientation at a ceramic grain boundary might cause a large change in energy by causing like charges to approach each other. Another factor which may limit the capacity of a ceramic grain boundary to act as a source is its affinity for foreign atoms. A concentration of foreign atoms in the boundary region could reduce its effectiveness as a source of dislocations by raising the local friction stress.

Further, the nature and amount of impurities at the boundaries can also be of great significance in other ways. Impurities can lead to the presence of essentially another phase as a film of varying thickness between the grains. Under these conditions the strength of the grain boundaries may not be adequate to transmit stresses and strains. Deformation or shearing then can occur along grain boundaries leading to separation. The presence of Li and F in the Type 1 specimens may be responsible in this way for their grain boundary shearing described earlier. The Type 2 specimens may, then, attribute their behavior to the presence of stronger and more coherent grain boundaries. Research efforts to correlate characterized grain boundaries with the behavior of polycrystalline pieces are still quite limited.

Carnahan has attempted to increase the strength of polycrystalline MgO at room temperature by adding a second dispersed phase of tungsten spheres.[49] It was reasoned that such spheres might produce mobile dislocations when the specimen was stressed as observed by Jones and Mitchell in the case of glass spheres in AgCl.[50] The increased number of sources would be expected to produce a more homogeneous distribution of slip and

thus raise the strength; however, no increase in strength was observed. No effort was made to directly establish the distribution of slip in the grains either with or without the tungsten spheres.

Another approach for producing a more homogeneous distribution of slip in a polycrystalline ceramic is suggested by the recent experiments of Bullen and coworkers on recrystallized chromium.[51] They report that recrystallized chromium, which is ordinarily quite brittle, exhibits considerable ductility if subjected to a hydrostatic pressure prior to but not during deformation. They attribute this behavior to the formation of mobile dislocation segments at internal inhomogeneities such as precipitates or inclusions. If this interpretation is correct, then it suggests that a homogeneous distribution of slip might be produced in a polycrystalline ceramic material by alloying followed by a heat treatment to form a suitably dispersed precipitate and finally by application of hydrostatic pressure.

C. Movement of Slip Systems

Another fact, which needs to be established in the case of polycrystalline MgO, is the role of cross-slip in relieving stress concentrations where slip bands intersect grain boundaries.* Such relief has been postulated by Stokes and Li to explain the slight ductility exhibited by NaCl bicrystals at room temperature and MgO bicrystals at 450°C.[6] Such relief would explain the lack of cleavage in the grains of the Type 2 specimen deformed at 400°C. It might be argued that the formation of cleavage cracks in this specimen was suppressed by the compression

*The term cross-slip in this case refers to the movement of screw dislocations with a/2 {110} Burgers vectors for short distances on {100} planes.

loading; however, as discussed in Section IV-C, high stresses were generated between grains in addition to the applied load. Since these stresses were sufficient to cause grain boundary parting, it would seem that cleavage cracks, if nucleated, should have been observed. Further information about stress relief by cross-slip might be obtained by careful study of crack nucleation by individual slip bands in bicrystals of MgO as a function of temperature.

The greatest barrier to promoting ductility in ceramic materials is their failure to satisfy the von Misés requirement for movement on five independent slip systems. This failure is intrinsic and can generally be related to the atomic structure at the core of the dislocation. In MgO and other predominantly ionic materials with the NaCl-type structure, Gilman has pointed out that the high stress required for slip on {001} $\langle 1\bar{1}0 \rangle$ slip systems is due to the close approach of anions at the dislocation core when it is in mid-glide position.[5] Such slip is less difficult in compounds with more polarizable anions or at higher temperatures where the lattice is expanded.

Kelly and Clarke have suggested that some form of alloying might be used to make the stress required for slip on the {001} $\langle 1\bar{1}0 \rangle$ slip systems more comparable to that required for {110} $\langle 1\bar{1}0 \rangle$ slip in MgO. The results of several investigations of the effect of alloying on NaCl-type compounds have not been encouraging, however, in this regard. Stoloff, Lezius, and Johnston studied the behavior of KCl-KBr alloys.[8] Since the Br^- ion is more polarizable than the Cl^- ion, a lowering of the stress required for {001} $\langle 1\bar{1}0 \rangle$ slip would be predicted with increasing Br^- ion additions. Instead, it was found that the Br^- ion additions suppressed slip on these slip systems. Liu, Stokes, and Li have investigated the

mechanical behavior of MgO-NiO and MgO-MnO alloys but observed no change in slip mode at room temperature.[53]

In addition to the problem of realizing movement on five independent slip systems to satisfy the von Mises requirement, there is the additional problem of intersections of slip systems which also must occur or be accommodated in some manner to obtain ductility. Resistance to such intersections has been discussed in the previous section. Efforts to minimize such problems and related effects by alloying or other means have not been reported.

D. Polycrystalline Material of Preferred Orientation

Although failure to satisfy the von Mises requirement precludes continuity in a deformed polycrystalline material with randomly oriented grains, it does not preclude continuity in a polycrystalline material with a preferred orientation which has undergone a specific change in shape. Consider, for example, the capacity of a polycrystalline specimen of MgO to undergo a uniaxial strain, if each grain is oriented so that a <100> axis is oriented parallel to the stress axis as shown in Fig. 19. Continuity can be maintained if each grain takes on the overall strain of the specimen, i.e., if in each grain

$$\epsilon_{33} = \text{uniaxial strain}, \qquad \text{(i)}$$

$$\epsilon_{11} = \epsilon_{22} = -\frac{1}{2}\epsilon_{33} \qquad \text{(ii)}$$

and

$$\epsilon_{12} = \epsilon_{13} = \epsilon_{23} = 0\,, \qquad \text{(iii)}$$

where the strain components are referred to axes 1, 2, and 3 in Fig. 19. If a grain is oriented so that its <100> axes lie parallel to the

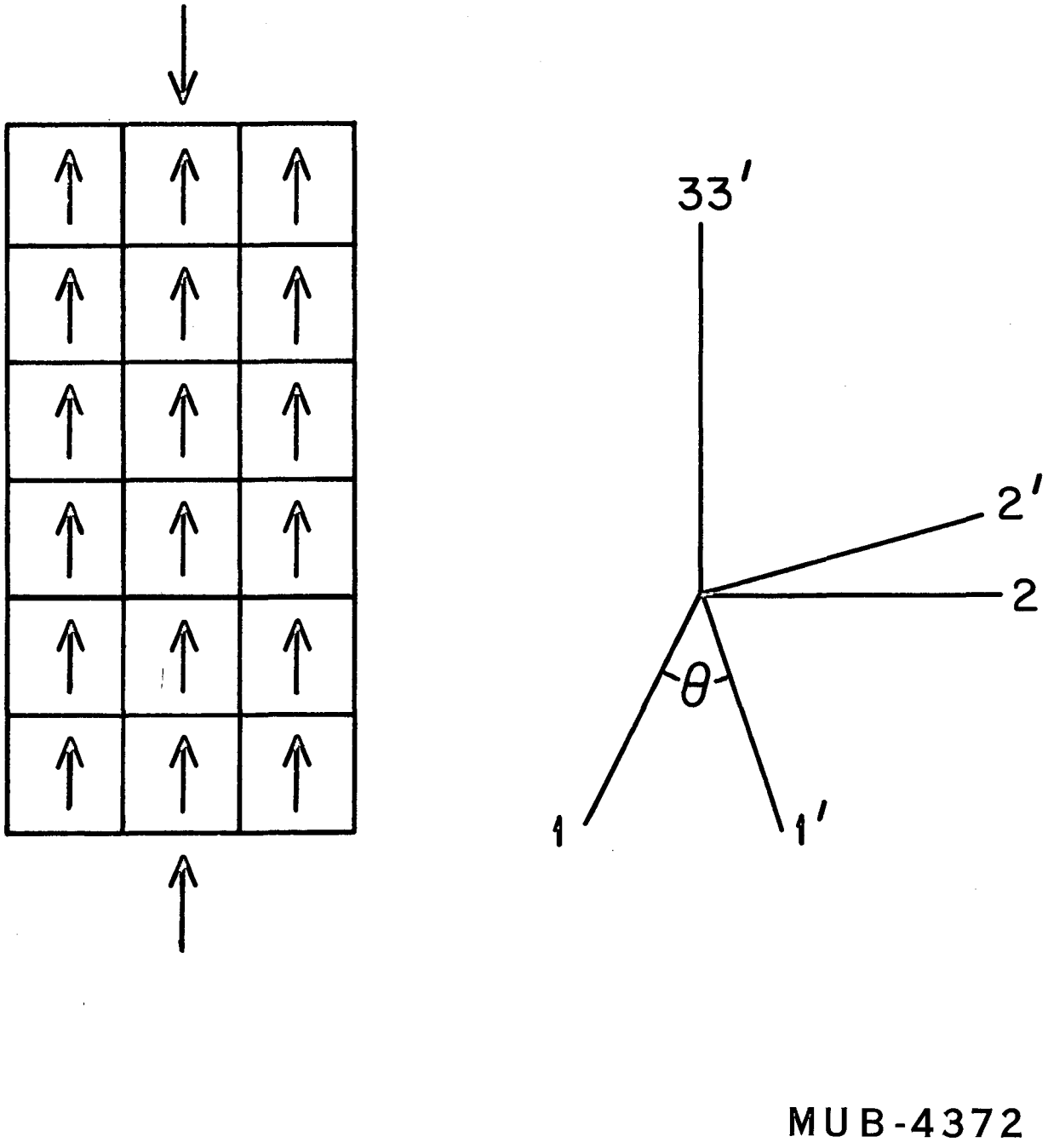

Fig. 19. Schematic sketch of an isoaxial compact.

(1, 2, 3) axes, equal amounts of slip parallel to the four <110> planes lying at 45° to the stress axis can produce the required strains. (This grain is the three-dimensional analog of the grain shown in Fig. 4b.) If a grain is oriented so that its <100> axes 2' and 3' are rotated by an angle θ about the stress axis, it can also change shape in the required manner. This result can be seen by expressing the strain components referred to the primed axes in terms of strain components referred to the unprimed axes. The two sets of strain components are related by the linear equation

$$\epsilon'_{\ell m} = \alpha_{\ell i}\, \alpha_{mj}\, \epsilon_{ij} \tag{8}$$

where

$$\alpha_{\ell i} = \hat{e}'_{\ell} \cdot \hat{e}_{i}\,, \tag{9}$$

$\{\hat{e}_{\ell}\}$ and $\{\hat{e}_{i}\}$ are unit vectors for the primed and unprimed systems, and the summation convention is to be applied. Expanding this equation we obtain

$$\epsilon'_{11} = \cos^2\theta\, \epsilon_{11} + \sin^2\theta\, \epsilon_{22} + 2 \sin\theta \cos\theta\, \epsilon_{12}$$

$$\epsilon'_{22} = \sin^2\theta\, \epsilon_{11} + \cos^2\theta\, \epsilon_{22} - 2 \sin\theta \cos\theta\, \epsilon_{12}$$

$$\epsilon'_{33} = \epsilon_{33}$$

$$\epsilon'_{12} = -\cos\theta \sin\theta\, \epsilon_{11} + \sin\theta \cos\theta\, \epsilon_{22} + (\cos^2\theta - \sin^2\theta)\epsilon_{12}$$

$$\epsilon'_{13} = \cos\theta\, \epsilon_{13} + \sin\theta\, \epsilon_{23}$$

$$\epsilon'_{23} = -\sin\theta\, \epsilon_{13} + \cos\theta\, \epsilon_{23}\,.$$

The strain components referred to the primed system which correspond to the required strains in the unprimed system may be found by substituting conditions (i)-(iii) into the preceding equations. The resulting strain components are

$$\epsilon'_{33} = \text{compressive strain}$$

$$\epsilon'_{11} = \epsilon'_{22} = -\frac{1}{2}\epsilon'_{33}$$

$$\epsilon'_{12} = \epsilon'_{13} = \epsilon'_{23} = 0$$

which as already discussed can be produced by equal amounts of slip parallel to the four {110} planes at 45° to the stress axis thus proving the assertion.

Finally, it is interesting to note than an isoaxial compact such as shown in Fig. 19 will exhibit an unusual anisotropy in grain boundary strength. If a uniaxial stress is applied perpendicular to that indicated in Fig. 19 and at 45° to the plane of the page, the maximum resolved shear stress falls entirely on tilt boundaries which according to the data of Murray and Mountvala presented in Section III-A are the most resistant to sliding. The resolved shear stress on the weaker twist boundaries is zero.

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