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Authors

Langdon, Terence G.

Pask, Joseph A.

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Terence G. Langdon* and Joseph A. Pask

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

I. INTRODUCTION

There is at the present time a great need for structural materials capable of withstanding both an oxidizing environment and also the very high temperatures at which many metals melt or become so ductile that they have only a limited practical application. Magnesia (MgO), with a melting point of 2800°C and general availability, is thus of considerable interest. This interest is enhanced by the fact that its NaCl-type crystal structure provides the potentialities of realizing some plastic deformation under applied loads, which is highly desirable for such applications. Of further interest is the fact that its isotropic crystal structure allows a polycrystalline MgO specimen to be transparent in the absence of any other phases, either pores and/or condensed phases. As a result there has been a considerable amount of work directed toward the development of an understanding of the mechanical behavior of single crystals and polycrystalline specimens of MgO. Because of the need for

* Presently at Department of Surface Physics, Cavendish Laboratory,
University of Cambridge, Cambridge, England.

polycrystalline specimens of theoretical density and with controlled character, there has been a parallel development effort extended in this direction.

The MgO crystal, or periclase, is an ionic crystal whose model structure can be considered to consist simply of hard spheres bound together by electrostatic attractive forces. The oxygen ions are close-packed according to a face-centered cubic (f.c.c.) arrangement. This packing has octahedral holes (coordinated by six oxygens) equal to the number of oxygens and twice as many tetrahedral holes (coordinated by four oxygens). The magnesium cations occupy all of the octahedral holes. The resulting NaCl-type structure is pictured in Fig. 1; it can then be considered to consist of two interpenetrating f.c.c. lattices, one of oxygens and one of magnesiums, as shown schematically in Fig. 2.

Ionic crystals of the NaCl-type deform by translation gliding, or slip, most easily on $\{110\}$ planes as shown in the upper sketch of Fig. 2. At high temperatures glide can also readily occur on $\{100\}$ planes (lower sketch of Fig. 2). Observations of slip on $\{111\}$ planes above 1600°C have also been reported; there is some doubt, however, as to whether slip can actually occur on these planes. In all cases the direction of gliding is $\langle 110 \rangle$ which is the shortest translation vector of the crystal structure, i.e., it connects nearest neighboring ions of the same sign as seen in Fig. 2. This is the direction of densest rows of like ions; it thus is the direction that requires the smallest amount of displacement across the glide plane to restore the structure. Also, no strong electrostatic repulsion forces occur between cations in the process of $\langle 110 \rangle$ gliding. It should also be pointed out that any gliding in the $\langle 100 \rangle$

direction would result in the juxtaposition of like ions with the development of large electrostatic repulsive forces.

Geometrically, however, it would be expected that glide on the $\{100\}$ $\langle 110 \rangle$ slip systems would be more favorable than on the $\{110\}$ $\langle 110 \rangle$ slip systems on the basis that the $\{100\}$ planes are more densely packed and have the wider spacing. The reason that they are not the primary glide planes is probably due to the fact that gliding on $\{100\}$ planes is accompanied by some electrostatic faulting (Gilman 1961); in gliding, the cations come close enough to each other at half the unit translation distance to develop strong repulsive forces. It can be seen by examining Fig. 2 that this does not happen in gliding on $\{110\}$ planes in the $\langle 110 \rangle$ direction. It is of interest to note that the tendency for glide on $\{100\}$ planes increases with increase in temperature; it also increases as the ionic character of the binding decreases, e.g., PbTe (NaCl-type structure) prefers to glide on $\{100\}$ $\langle 110 \rangle$ slip systems at room temperature. It should be noted that in ionic NaCl-type crystals the $\{100\}$ planes are cleavage planes; this characteristic may be associated with any distortion in the $\langle 100 \rangle$ direction. Although geometrically most favorable on the basis of density and spacing, it appears that glide on $\{111\}$ planes would be inoperable because it would result in offsets at the faces of the crystal either with an excess or a deficiency of positive charges and thus produce an electrostatic unbalance.

Uniform instantaneous slip over an entire given plane is literally impossible because of the large amount of energy that would be required to break all of the bonds in the process of flow. Flow mechanisms become possible because of the presence or introduction of mobile line

defects, known as dislocations, in the crystal. These can move by breaking a relatively small number of bonds at any given instant. There have been many papers in this subject area; two reviews that provide a good introduction to the subject of dislocation theory have been written by Gilman (1961) and Washburn (1967).

As mentioned, in MgO easy glide occurs on the $\{110\}$ $\langle 110 \rangle$ family of slip systems. In this family there are three orthogonal pairs of slip planes in each of which the Burgers direction vectors shown at the right in Fig. 3, are also at right angles. The angles formed between Burgers vectors from different orthogonal pairs of slip planes are oblique (120°). It can be shown that the resolved shear stress on each slip system of an orthogonal pair is equal for any uniaxial stress. Also, because of the random orientation of grains in a polycrystalline piece, it is desirable to know the shear stresses developed on all potential glide planes for any orientation of a grain. This analysis has been presented by Hulse et al. (1963). In the $\{100\}$ $\langle 110 \rangle$ family there are also six slip systems consisting of three planes at right angles, with each plane having two slip directions at right angles to each other which are the same as those shown at the right in Fig. 3. The angles formed between Burgers vectors from two slip planes are again oblique. Traces of all the $\{110\}$ and $\{100\}$ planes are shown in the left sketch of Fig. 3.

The following criteria must be fulfilled in order to realize uniform plastic deformation by slip in a polycrystalline matrix: there must be an adequate number of independent slip systems, homogeneous slip must occur throughout a crystal, and the different crystallographic slip systems must have the ability to interpenetrate. Unfortunately, at lower

temperatures these criteria are not fulfilled. Limited slip frequently leads to the nucleation of flaws which result in cleavage fracture. It thus becomes as equally important to study dislocation behavior to realize reliable high strengths as it is to realize plasticity. These factors and preceding items will be discussed and developed.

Although several review articles on mechanical properties have been published in the past (Wachtman 1957, Gilman 1961, Stokes and Li 1963a, Miles 1964a, Stokes 1965b, 1966a, Wachtman 1967), these have endeavored to cover the complete range of ionic crystals; in this chapter, it is intended to review in some detail those investigations which refer specifically to MgO.

II. SINGLE CRYSTALS

It is only within the last decade that any detailed investigation of the mechanical properties of ionic crystals has been undertaken. This apparent omission arose because ceramic materials were for many years thought to be inherently brittle. It was not until 1958 that a group of investigators at the University of California, by applying dislocation theory as obtained from a study of metals, predicted that ionic solids having cubic crystal structures would possess a reasonable degree of ductility even at ordinary temperatures.

The original tests, carried out on freshly cleaved single crystals of sodium chloride and potassium chloride (Gorum et al. 1958, Parker et al. 1958), showed that ductility was possible, and that the crystals could be bent by hand but for only less than a minute after exposing the cleavage faces to air. A similar ductility was also observed in magnesium oxide at room temperature, using small pieces cleaved from an

impure single crystal. In these tests, strains of up to 20% were realized on the tension side of a bend-test specimen, although it was again found that the ability to bend the MgO crystals decreased with the time after cleaving; a similar observation was also reported by Lad et al. (1958). In later work (Gorum et al. 1960), the effect of a high temperature (2000°C) heat treatment was examined, and it was found that this made a specimen either weaker and more ductile or stronger and more brittle depending on whether the subsequent cooling was fast or slow. Purity was also an important parameter, since crystals containing only 10 ppm of Fe flowed at a much lower stress than those containing 30 ppm or more. Much of this early work on MgO single crystals was devoted to determining the mechanical properties at room temperature, and it is convenient to first review this data before examining the results obtained when the temperature is increased.

From tension tests, experimental evidence was presented by Washburn et al. (1959) (see also Parker 1959, 1961) to support the theory (Cottrell 1958) that cleavage fracture can be initiated by the nucleation of cracks on {100} planes due to the coalescence of dislocations on two intersecting {110} slip planes. It should be noted, however, that since the cracks did not appear to form until the slip bands had reached a certain critical width, a modified method of nucleation has also been suggested (Orowan 1959, Argon and Orowan 1964b - see also Briggs et al. 1964, Briggs and Clarke 1965). Tests in three-point bending by Washburn et al. (1959) revealed that one slip system frequently dominated on the tension side of the beam, with the conjugate system operating on the compression side. Since the specimens were loaded such that four of the

{110} <110> slip systems were subjected to the same resolved shear stress, it was concluded that dislocations on a slip band in one system were effective barriers to slip on an intersecting system. Such lack of interpenetration, which is considered in more detail later, was initially proven by the observation that dislocations on an intersecting system could pile up against a barrier imposed by the initial slip to the extent of bending the crystal through greater than 3° , as revealed by the direction of slip bands on the two sides of the barrier. The actual mechanism of slip band intersection has been analyzed in some detail by Argon and Orowan (1964a). Washburn et al. (1959) also observed that the grown-in dislocations did not move during plastic deformation, and did not act as effective barriers to dislocation motion. The significance of these grown-in dislocations will be discussed in more detail later.

In a detailed study of the formation of cracks in MgO single crystals tested in compression, Stokes et al. (1958) confirmed that slip took place over {110} planes in <110> directions, and found that cracks originated in kink bands which were produced as a result of non-homogeneous compression. If the cleaved surface was sprinkled with silicon carbide particles, slip dislocations were generated by impact, and these could be revealed as rosettes on the surface by means of a suitable etch, as shown in Fig. 4 (after Pask and Copley 1963). On loading a sprinkled crystal, slip then occurred with each slip line passing through a rosette; a typical example is shown in Fig. 5. Direct support for the Stroh mechanism of crack formation (Stroh 1954, 1955, 1957), whereby a crack is nucleated as a result of the stresses associated with the pile-up of edge dislocations against a barrier, was obtained by compressing

crystals to a strain of about 3%. These "Stroh cracks" lay on conjugate $\{110\}$ slip planes, instead of the usual $\{100\}$ cleavage planes, and extended right through the crystal as tiny slits in a $[001]$ direction. Secondary cracks were also observed, but these were tapered and did not extend through the crystal. To explain these observations, it was noted that Stroh cracks were nucleated by the pile-up of dislocations against the kink bands, as shown in Fig. 6. Once formed perpendicular to their associated slip plane, the cracks quickly ran into the compression stress field of the next pile-up, thereby restricting their total length but allowing them to spread rapidly along the length of the edge dislocation line to form a $[001]$ direction slit. Furthermore, they were able to increase their length by advancing into the region within the kink band to form a secondary crack.

The formation of Stroh cracks was later confirmed by Johnston (1960), and Stokes et al. (1959a) showed that they were frequently observed lying parallel to the (110) slip planes in both the regions of compression and tension after simple bending. It was also observed, by etching, that the slits developed in three-point bending were confined between two adjacent orthogonal $(1\bar{1}0)$ slip bands (Stokes et al. 1959c). An example of the slits associated with the intersection of orthogonal slip bands is shown in Fig. 7; the wavy lines visible here are cleavage steps, and can be disregarded. Stokes et al. (1959c) also noted that the cracks could not only be nucleated where slip bands intersected, but more important, they could be stabilized by meeting a slip band. This observation is of particular importance, since it shows that if micro-cracks form in the early stages of plastic flow, when there is little slip present, their

growth is unimpeded and the material is brittle. If, however, the crystal is first plastically deformed to introduce a sufficient number of slip bands, the growth of the micro-cracks is restricted in certain directions and they develop into narrow slits which do not lead immediately to catastrophic failure. Thus it is the growth, and not the nucleation, of the micro-cracks which is the critical stage in the fracture process, and this is determined primarily by the density and distribution of slip.

An alternative type of crack nucleation was suggested by Keh et al. (1959), whereby a crack forms on the (110) slip plane due to the coalescence of dislocations by intersection of the (011) and (101) slip planes at an angle of 120° . Experimental evidence for this type of crack was obtained from hardness indentations (Keh et al. 1959, Keh 1960), and similar cracks have also been reported by Atkins and Tabor (1967) from a mutual indentation technique.

The surface condition of MgO single crystals is extremely important in determining the mechanical behavior. For example, etch-pit studies, carried out in bending and tension, have revealed that the first few dislocation loops moving on the (110) planes originate from sources located at the surface (Washburn and Gorum 1960). A detailed examination of the effect of surface condition was reported by Stokes et al. (1959b), in which it was found that, although the yield stress of crystals sprinkled with silicon carbide particles was identical to that of unsprinkled crystals from the same source, there was a marked difference in the observed dislocation behavior. In the sprinkled crystals, the "fresh" dislocation half-loops introduced by the sprinkling started to expand at approximately one-third of the "yield stress," and multiplication

commenced above about one-half of this; in the unsprinkled crystals, however, slip was not detected until two-thirds of the "yield stress," which was the point at which the impact half-loops underwent gross multiplication. It was suggested, in a study of the effect of surface micro-cracks (Stokes et al. 1960), that the appearance of the fracture surface was the most reliable criterion for determining whether fracture was due to the presence of surface flaws. Thus, when crystals were tested with a surface which had been "damaged," whether by mishandling or in cleavage, the cleavage lines at the source of fracture radiated from a point located on the periphery of the fracture surface, and this point coincided with a surface flaw. In polished crystals, however, the fracture nucleated as a direct consequence of the dynamic interaction of orthogonal slip bands, as first reported by Washburn et al. (1959). It was also pointed out that it is not essential to chemically polish single crystals in order to obtain a surface free of micro-cracks, since this can be attained by cleaving MgO on a soft pad of tissue paper. The effect of surface condition has also been reported by Bruneau and Pratt (1962) for tests in bending.

Clarke and Sambell (1960) showed that surface defects may exist in the form of micro-cracks at the crystal edges which, although at least an order of magnitude below the critical Griffith size, are still able to grow after slip has occurred, and thus lead to fracture. It was also found that if the cracks were removed by polishing, the ductility was enhanced five to ten times. In a later study (Clarke et al. 1962b), the dislocation movements associated with the growth of micro-cracks in cleaved single crystals were revealed by recording the changes in stress

birefringence on cine-film. A mechanism was suggested to account for the growth of these micro-cracks, and this is illustrated schematically in Fig. 8. In this model, a micro-crack ABC exists at the edge of the crystal, and slip spreads out from point B in the manner depicted. New dislocation sources are formed in the body of the crystal by double cross-slip, and this gives a band of slip of which a part passes under the micro-crack front (such as the edge dislocations along line EF). Although the diagram shows that the screw components have cross-slipped onto a plane above the initial plane BG, cross-slip to a lower plane can also occur. There is a strain field associated with the pile-up of dislocations on intersecting systems at F, and this supplements locally the strain due to the applied load, and thus favors the growth of the micro-crack along FH. This growth is very slow until the micro-crack reaches the critical Griffith size, and it is possible to reduce the load while the crack is growing and thus prevent complete failure of the specimen (Tattersall and Clarke 1962).

The tensile behavior of chemically polished crystals at room temperature was examined in detail by Stokes et al. (1961), and it was found that the fracture behavior depended critically upon the relative orientation, number, thickness and spacing between the slip bands. Thus a crystal was completely brittle if two slip bands, generated on orthogonal $\{110\}$ planes, happened to intersect to nucleate a crack before other slip bands had developed. Ductility was possible, however, if there was a high density of slip sources, and the cracks could be stabilized by the adjacent slip bands. If, in the extreme case, slip was confined solely to a single slip band, then this expanded laterally to fill the entire

gauge length and the crystal was extremely ductile. These three possibilities are depicted in Fig. 9, wherein the yield stress remains consistently in the range 4000-5000 p.s.i., but the elongation at fracture varies from less than 0.2%, so that the specimens appeared completely brittle, to in excess of 7% (the total possible elongation for the type III specimens is not known since, unlike the types I and II which always fractured within the gauge length, these specimens failed by pulling loose from or fracturing at the grips). As shown in Fig. 9, there is some evidence of a yield point in the type III crystals. An examination of specimens of type II showed that they contained cracks at relatively small strains, but these were stabilized by the density of slip sources and thus the crystal continued to deform. Although the reason for the variation in the number of intrinsic slip sources operative from the onset of plastic flow for nominally identical crystals was not fully understood, it was found experimentally that crystals originally prepared from the same bulk crystal usually fell into the same category (I, II or III); this suggests that the amount and distribution of impurities is an important factor.

Further work by Stokes et al. (1961) on crystals with cleaved surfaces showed that these were usually of type II, since slip sources were introduced by the cleavage process, and an optical examination of the specimens after fracture revealed many stable (110) [100] slits of the type described previously. These results suggest that if the number of slip sources is increased substantially by sprinkling, the distribution of slip bands would be sufficiently dense to suppress the formation of cracks by the conventional mechanism. In fact, tests in tension showed

this was not the case since, although the sprinkled crystals were always ductile due to the high density of slip artificially introduced, the gauge length became subdivided into a number of blocks within which slip was very dense and confined to a single slip plane. The reason that slip should be confined in this manner, although many slip sources were present on all four equally stressed planes, was not clear, although it was suggested that it may be a consequence of a non-uniform stress distribution due to misalignment or variation in cross-section along the gauge length. The result of this block formation, however, was that slits were confined entirely to the regions of less dense interpenetrating orthogonal slip located at the boundary between two adjacent blocks.

In bending, the situation was somewhat different since the crystals always slipped on the two $\{110\}$ planes having the bend axis as their zone axis, and sprinkling then gave a three to five-fold improvement in ductility. Unlike tension, no slip blocks were formed in this case, and stable $(110) [001]$ slits did not occur. It appears that this may be due either to the possibility that sprinkling reduces the spacing between slip bands below microscopic dimensions, so that slits, if formed, are not optically visible, or because sprinkling introduces a high number of slip sources which effectively homogenizes the strain.

The results in tension support the previous suggestion that stabilization of a slit is possible (Stokes et al. 1959c). For the mechanism of slit nucleation, however, it was noted that a slit was formed not when a slip band burst across the crystal and was held up by an intersecting slip band, thereby resulting in a sudden avalanche of edge dislocations piling into the obstacle, but rather when two slip bands had

completely cut through each other. This is illustrated schematically in Fig. 10 in which two independent slip bands (Fig. 10a) grow in width to meet and intersect one another (Fig. 10b), and a crack can nucleate (Fig. 10c) by the piling up of dislocations moving in band Y against the barrier provided by band X. After nucleation, the crack can then propagate in the [110] direction along the edge of slip band X, and then in the [100] direction across the band. The development of the fracture path is shown in Fig. 10d with A being the point of nucleation, from which it is seen that slip band Y continues to grow in width until it is finally cut off by the fracture. A composite photomicrograph showing this effect is given at the bottom of Fig. 10.

The investigation just described emphasizes that the yield stress is dependent chiefly upon the strain rate and not at all upon the number of "fresh" dislocation sources originally present; this is true even if a crystal contains only one "fresh" source since prolific dislocation multiplication is able to take place at the edge of the slip band, and the crystal then shows the same yield stress and rate of work hardening as in a sprinkled crystal. The question now arises, however, of the status of crystals containing no "fresh" sources. To investigate this problem, Stokes (1962) chemically polished crystals for 30 minutes to remove all "fresh" sources, and then loaded them with extreme care and tested in tension. These crystals, which contained only the grown-in dislocation sources, were found to deform entirely elastically up to stresses of 30,000-50,000 p.s.i., after which they yielded with a sharp drop in stress down to 8000-9000 p.s.i., and then deformed plastically with a number of intersecting slip planes visible in the gauge length.

It is interesting to note that the stress of ~ 8000 p.s.i. corresponds closely with the stress level at which the "fresh" sources were observed to operate in the sprinkled crystals. However, whereas a specimen with a "fresh" source yields with a smooth expansion of a single slip band, the slip in specimens with only grown-in sources fluctuated between a number of intersecting planes. This is probably due to the much higher stress level at which the latter source begins to operate, since the initial burst of dislocations must move with a very high velocity, due to the high stress; and the movement of this concentrated stress pulse through the lattice is thus able to trigger off other grown-in sources. While these results refer specifically to tensile tests, experiments were also conducted in bending but it was then found that there was an unavoidable introduction of "fresh" dislocations by mechanical contact along the three loading beams.

These results suggest that the grown-in dislocation sources are associated with impurity precipitate particles, and direct evidence of this will be presented later. It was found that these particles could be dissolved by annealing at 2000°C , so that the crystal was essentially free of dislocation sources and was then consistently stronger. For example, Stokes and Li (1963b) found that annealed and polished crystals deformed elastically up to stresses in excess of 160,000 p.s.i.; however, if a crystal was annealed at 2000°C , cooled, and then sprinkled, it yielded at the lower stress level again, and was highly ductile. It should be noted that Alden (1963a, 1963b) has achieved maximum stresses in tension of up to 112,000 p.s.i., by applying an alternating stress to sprinkled crystals containing a high density of dislocation sources.

Keh (1960) showed that it was also possible for nominally "fresh" dislocations to be pinned by a high temperature anneal, by introducing dislocations on one surface of a cleaved crystal with an indenter, annealing at 1000°C , putting several more indentations on the opposite surface and etching to show the "fresh" and "aged" dislocations, and then stressing in compression until macroscopic yielding occurred. On re-etching, it was found that most of the glide bands were initiated from the "fresh" dislocation rosettes, while none was initiated from the "aged" ones.

It is clear from these results that the dislocations of the grown-in network do not move at all, for if they moved only a short distance then "fresh" dislocation loops would be nucleated by the cross-slip mechanism and yielding would immediately occur. As it is unlikely that the dislocations are firmly pinned by the impurity precipitate particles, since bowing between the pinning points could still occur, it appears instead that the grown-in dislocations lie on the wrong slip plane or possess the wrong Burgers vector to be mobile at room temperature. Since it is only dislocations with $a/2[110]$ Burgers vectors lying in the $\{110\}$ slip plane which can move easily without causing electrostatic faulting (Gilman 1959), it is possible that these are, for example, $a/2[110]$ dislocations lying in the closely packed $\{100\}$ planes. Furthermore, $a/2[110]$ dislocations lying in other planes will be even less mobile, and dislocations possessing Burgers vectors other than $a/2[110]$ can only move by introducing considerable electrostatic faulting into the crystal. In contrast, "fresh" dislocation sources have the correct $a/2[110]$ Burgers vector, they lie in the most mobile $\{110\}$ plane, and are completely free of contamination by impurities.

While the results discussed so far have dealt specifically with investigations carried out at room temperature, a large volume of work has also been published on the high temperature mechanical properties of MgO single crystals. Hulse and Pask (1960) deformed cleaved crystals in compression with a $\langle 100 \rangle$ loading axis, using a constant rate of loading of 20 psi/sec., and obtained a plot of the bulk yield stress versus temperature in the range -196°C to 1200°C . This plot decreased exponentially with increasing temperature, until it became essentially constant at about 900°C . This trend was also noted by Sinha (1964), and a similar decrease was reported by Thompson and Roberts (1960) from four-point bending tests in the range -148°C to 507°C , although in the latter case marked differences were observed in specimens cleaved from different parent crystals. In the work of Hulse and Pask (1960), all crystals work hardened after yielding, the rate increasing gradually with strain except at 1200°C where it remained constant until failure. The deformation patterns were revealed by etching, and this showed that slip on one set of (110) planes had difficulty in passing through slip bands formed on the conjugate set of (110) planes, so that there were appreciable areas where multiple slip did not occur. Dew-Hughes and Narlikar (1967) also carried out compression tests on crystals with a $\langle 100 \rangle$ loading axis, at room temperature only, and under conditions of constant strain rate. They reported a linear, but low, rate of work hardening immediately after yielding, up to 0.7-1.5% strain, after which the rate was again linear but very much increased. Since examination of the deformed crystals by X-ray microscopy (Narlikar and Dew-Hughes 1962-63) showed that they had become fragmented into many small crystallites during this second stage,

it was suggested that the change in rate was due to the pile-up of dislocations against these sub-grain boundaries.

An apparent anomaly should be noted here which undoubtedly is the effect of the method of testing. Hulse and Pask (1960) with a constant loading rate observed the strain-hardening rate, as measured by the slope of the first approximately linear portion of the stress-strain curve, to increase with increasing temperature in the range 26°C to 1030°C (see Copley and Pask (1965b) for plot). However, Phillips (1960), working in compression with a similar orientation but with a constant strain rate, found the rate of work hardening, as measured by the slope of the stress-strain curve at 4% strain, to sharply decrease with increasing temperature in the range 25-1000°C. Later work (Phillips 1962), taking the slope of the stress-strain curve at 1% strain, showed a sharp decrease over the temperature range -203°C to 600°C.

Single crystals were also tested in compression with both $\langle 110 \rangle$ and $\langle 111 \rangle$ loading axes (Hulse et al. 1963), covering the temperature range 26°C-1250°C. A plot was obtained of yield stress versus temperature for these loading axes, and this was compared with that obtained previously for the $\langle 100 \rangle$ loading axis (Hulse and Pask 1960). Two of these curves, for $\langle 100 \rangle$ and $\langle 111 \rangle$ loading axes, are shown later (Fig. 29) where they are compared with results for polycrystalline specimens; the other curve, for the $\langle 110 \rangle$ loading axis, is intermediate between these two. In analyzing these results, it is necessary to consider the effect of the orientation of the stress axis. Thus, specimens with a $\langle 100 \rangle$ stress axis, which are easily obtained by cleavage, have a resolved shear stress equal to one half of the applied stress acting on four of the six

{110} <110> slip systems wherein in those with a <110> stress axis the resolved shear stress still acts on four of the six {110} <110> slip systems but is now equal to only one-quarter of the applied stress. In both orientations, the resolved shear stress acting on the remaining two slip systems is zero. It is therefore to be expected that the applied uniaxial stress needed to yield crystals with a <110> loading axis should be twice that required to yield crystals with a <100> axis, assuming that in both orientations slip takes place solely on the {110} <110> slip systems. The results showed reasonable agreement with this analysis, although with a systematic deviation below 800°C which, it was suggested, probably arose through the use of crystals from different batches for the two series of tests. When specimens are tested with a <111> loading axis, no resolved shear stress is realized on any of the {110} <110> slip systems, and etching revealed that the specimens then deformed plastically on the {100} <111> slip systems at temperatures above 350°C. The very high yield stresses observed for the <111> loading axis arise because of the strong electrostatic repulsive forces which must be overcome before dislocations of a/2<110> Burgers vector are able to move on {100} planes (Gilman 1959). This repulsive force, however, is less severe than that experienced in a <100> direction. As the temperature is increased, the crystal lattice expands and this leads to easier movement and a consequent reduction in the observed yield stress. While these results were obtained for crystals having constrained ends, similar values of yield stress have also been reported for crystals tested with a <111> loading axis with unconstrained ends (Hulse 1967).

Although the specimens of Hulse et al. (1963) explosively disintegrated into powder at a high stress level with a constant loading rate at room temperature, with no plastic yielding, an extrapolation of their data for a $\langle 111 \rangle$ loading axis suggests that the critical shear stress for (100) slip at room temperature will be extremely high. Weaver (1967) has recently compressed crystals of this orientation at a constant strain rate at room temperature, using a high confining pressure to prevent fracture. Even so, the stress for (100) slip was still not reached, and the observed behavior was markedly different from that reported by Hulse et al. (1963). At high pressures, the crystals deformed elastically to high stresses and then yielded abruptly with a sharp drop in stress. (It should be noted that at temperatures of 350°C and above, Hulse et al. (1963) using a constant loading rate observed a high rate of work hardening beyond the yield point.) An examination of the crystals deformed at room temperature showed that kinking had taken place on the dodecahedral (110)[110] slip systems, despite the fact that these are normally unstressed while three of the (100)[110] slip systems are under stress. Weaver (1967) showed, however, that only small local misorientations of the crystal, of the order of 2°, are needed to throw an appreciable resolved shear stress on the weaker (110)[110] slip systems. This work therefore serves to emphasize the importance of perfection of alignment when testing crystals with a $\langle 111 \rangle$ loading axis at low temperatures.

Copley and Pask (1965b) extended the earlier work of Hulse et al. (1963) to higher temperatures by compressing single crystals with $\langle 100 \rangle$ and $\langle 111 \rangle$ loading axes with constant loading rates at temperatures in the range 1000°C to 1600°C. Typical stress-strain curves for these two

loading axes are shown in Figs. 11 and 12. Of particular interest for the $\langle 100 \rangle$ stress axis is the change in shape of the curves above about 1100°C with the appearance of a gradual yield. The yield stresses for $\langle 111 \rangle$ orientated specimens, which were considerably higher than for $\langle 100 \rangle$ orientations at all temperatures, continuously decreased up to 1600°C . The complete plot of yield stress versus temperature, combining these results with those obtained earlier by Hulse et al. (1963), is shown later in Fig. 29. A $\langle 100 \rangle$ orientated crystal strained 14.8% at 1400°C is shown in Fig. 13a, and the same specimen is shown under polarized light in Fig. 13b. The birefringence visible here is a result of elastic distortions in regions where the density of dislocations of one sign is high; this serves to reveal the great localization of dislocation damage. The vertical striations visible in Fig. 13a on the upper part of the front face are associated with deformation bands which result from the separation of regions that have deformed by slipping on planes at 45° to the crystal surface (bulging bands) from those that have slipped on planes at 90° (flat bands). It was suggested by Copley and Pask (1965b) that the boundaries separating these two regions were formed by dislocations moving on oblique planes with Burgers vectors enclosing an angle of 120° , according to the reaction

$$\frac{a}{2} [\bar{1}01] + \frac{a}{2} [01\bar{1}] = \frac{a}{2} [\bar{1}10] \quad (1)$$

as first suggested by Kear et al. (1959). The segments of edge dislocation thus formed lie along the $[\bar{1}1\bar{1}]$ direction and, since they can only move in the $(11\bar{2})$ planes, are sessile.

This investigation also showed that, even at the highest test temperature of 1600°C , slip in any given region only occurred on two slip

planes at 90° to each other. Thus slip on one such orthogonal set of slip systems is seen to block slip on the other orthogonal set, even though both sets experience the same resolved shear stress. The 60° intersections are therefore considerably more difficult than those at 90° . Calculations were made, using the estimated number of etch pits formed in an edge dislocation band (Washburn and Gorum 1960), to show that the stress-strain curve obtained at room temperature could be satisfactorily explained in terms of the stress dependence of the dislocation velocity. This suggests that the decrease in yield stress for $\langle 100 \rangle$ stressed specimens with increasing temperature was due to an increase in the dislocation mobility. It was also postulated that the observed decrease in the strain-hardening rate at temperatures above 1100°C was due to the annealing-out of dislocation dipoles, owing to the increased diffusivity rates at the higher temperatures.

Impact tests were used by Johnston et al. (1959, 1962a) to study the brittle-ductile transition, and this revealed that it was possible to cleave MgO over (100) planes without macroscopic deformation at temperatures up to almost 2000°C ($\sim 0.7 T_m$, where T_m is the melting point in degrees Kelvin). This observation shows clearly that the ability of an ionic solid to accommodate plastic flow without fracture is strongly dependent upon the imposed plastic strain rate, since MgO single crystals may be bent at room temperature ($< 0.1 T_m$) at strain rates of $\sim 10^{-5} \text{ sec}^{-1}$, but under impact it is necessary to go to temperatures greater than 2000°C to accommodate similar deformation. It therefore follows that the observed brittle-ductile transition temperature is strongly dependent on the method of testing; for example, temperatures of $200\text{-}800^\circ\text{C}$, depending

on both time of air aging and source of material, were reported by Murray (1960) from tests in slow bending, a transition temperature of 850-1050°C was reported by Atkins and Tabor (1967) from mutual indentation techniques, and Sinha (1965) refers to a transition in bending at ~800°C.

The formation of kinks under bending, and the tendency for this to lead to fracture, was investigated by Stokes et al. (1962), and it was found that the tendency for fracture diminished as the temperature was increased. At high temperatures and low strain rates, the crystals were found to be ductile since the dislocations in the kink boundaries were no longer effective barriers to slip.

A detailed investigation of the mechanical behavior of MgO single crystals with a $\langle 100 \rangle$ loading axis was carried out by Day and Stokes (1964), covering a temperature range of 1000°C to 2000°C. Unlike the work of Hulse et al. (1963) and Copley and Pask (1965b) where specimens were tested in compression, these crystals were pulled in tension after annealing at 2000°C and chemically polishing. The results showed that the behavior was determined by the number of slip systems operating concurrently in any given volume, and three different situations arose depending on the temperature:

(1) At low temperatures ($\langle 1300^\circ\text{C} \rangle$), slip was confined to a single $\{110\} \langle 110 \rangle$ system since slip on any one $\langle 110 \rangle$ system was unable to penetrate slip on any other. The overall plasticity was therefore limited by the stress concentration which developed where slip switched from one plane to another. Generally, the slip alternated between two conjugate $\langle 110 \rangle$ systems within the gauge length, as shown by type I in

Fig. 14. Slight knots were formed, as indicated, at the points where slip switched from one conjugate plane to another. At these low temperatures, the specimens fractured in a brittle manner with cleavage frequently originating in the boundaries between two adjacent areas of slip on different planes.

(2) At intermediate temperatures (1300°C - 1700°C), $\{110\}$ $\langle 110 \rangle$ slip systems at 90° could intersect each other, but those at 60° could not. The mechanical behavior then depended on the initial slip distribution, and two possibilities arose. Firstly, there was the situation where slip originated throughout the whole gauge length on just one pair of interpenetrating conjugate $\langle 110 \rangle$ systems. Under these conditions, the specimens necked down to zero in one dimension only, as shown by type II (a) of Fig. 14. Secondly, there was the situation where slip switched from one pair of conjugate $\langle 110 \rangle$ systems to the other at some point along the gauge length. Since 60° intersections were not possible at these temperatures, the crystal divided into two blocks such that one block reduced in one dimension and the other in the second dimension, as shown by type II (b) of Fig. 14. Although there was some necking in the separate blocks, the specimens then fractured in a brittle manner by cleavage.

(3) At high temperatures ($>1700^{\circ}\text{C}$), the dislocations could interpenetrate on all slip systems, both at 60° and at 90° , although it was generally observed that slip was confined primarily to a pair of conjugate $\langle 110 \rangle$ planes within any given region. There was also evidence of slip on planes other than $\{110\}$ where the two systems switched over. This lead to a modified knife-edge fracture, as shown by type III in

Fig. 14, with a reduction in both dimensions, and recrystallization with a strong preferred orientation within the necked region. X-ray observations showed that extensive polygonization took place throughout the crystals at these temperatures, and at 2000°C the specimens elongated homogeneously by as much as 150% before the ductile fracture commenced.

It should be noted that, although these three differing types have been presented as occurring within certain discrete temperature ranges, this is not strictly correct since the temperature of transition between one type and another is dependent on the strain rate. At 1550°C, for example, each mode of behavior could be observed for different strain rates: type I at ~ 1.0 " min⁻¹, type IIa at ~ 0.1 " min⁻¹, type IIb at ~ 0.01 " min⁻¹, and type III at ~ 0.002 " min⁻¹. It is clear from these detailed results that a rise in temperature not only aids the mobility, and multiplication, of dislocations on the primary {110} planes, thereby reducing the yield stress, but also allows interpenetration of the slip systems so that the specimens become less brittle. The latter behavior is undoubtedly associated with a reduced tendency for cleavage.

The various observed transitions were considered in detail by Day and Stokes (1964b) in terms of the dislocation behavior. For intersection between the conjugate slip systems, the dislocation interaction must involve only their mutual cutting at a point, and it is therefore necessary to consider the interaction between screw dislocations (S_1) in the one plane with edge (E_2) or screw (S_2) dislocations in the other, as shown in Fig. 15 a and b respectively. In both cases a jog (J) is produced, which is constrained by its Burgers vector and direction to move in the cross-hatched {100} plane. Since the jog on S_2 cannot move forward

with the screw dislocation conservatively, a cusp is formed, and eventually an edge dislocation dipole which can be pinched off by cross-slip of the two separated segments of S_2 (as indicated by the arrows in Fig. 15a) to form a prismatic loop or "debris" in the slip plane. Cusps on screw dislocations after deformation at temperatures up to 1000°C have been observed using transmission electron microscopy (Groves and Kelly 1963b, Elkington et al. 1963) and "debris" has been reported in the slip bands up to 1200°C (Groves and Kelly 1962, 1963b). In the case of the jog on the edge dislocation, movement is confined to the $\{100\}$ plane and is therefore restricted due to electrostatic faulting (Gilman 1961). This may ultimately give rise to a screw dislocation dipole which can dissipate by cross-slip.

The temperature effect arises since the jogs are relatively immobile at low temperatures, and the debris causes further tangling, as observed in the electron microscope (Washburn et al. 1960a), and leads to the inability of two conjugate slip systems to interpenetrate extensively. As the temperature is raised, however, jogs in the edge dislocations become more mobile due to the increasing ease of slip over $\{100\}$ planes (Hulse et al. 1963) and the prismatic loops diffuse to form large single loops which can move under stress by a conservative climb mechanism (Kroupa and Price 1961). Thus the intersections are kept relatively free of debris, and interpenetration of the two conjugate slip systems increases with increasing temperature.

The situation is somewhat different for intersections between two slip systems making 60° angles (or 120°) to each other, since dislocations can then react according to the equation

$$\frac{a}{2} [110] + \frac{a}{2} [0\bar{1}1] = \frac{a}{2} [101] \quad (2)$$

This gives a drop in strain energy, and the formation of a third dislocation lying parallel to $\langle 111 \rangle$, as shown in Fig. 16a. This dislocation is pure edge and is restricted to moving in the $\{112\}$ slip plane, so that this dislocation may be regarded as sessile at low temperatures; direct observations of this reaction have been reported using electron microscopy (Groves and Kelly 1963b, Washburn et al. 1960a). A high density of these pure edge sessile dislocations are formed over the broad interface where the slip bands intercept, and these act as a barrier to flow at low temperatures. Above about 1300°C, however, they can rearrange into vertical polygonized arrays by climb, giving the observed longitudinal surface kinks. The result is a kink boundary lying in the $\{110\}$ plane, of the form shown in Fig. 16b. It is considered that these sessile dislocation walls are relatively immobile below 1700°C, constituting a barrier to interpenetration on two 60° slip systems, but above this temperature (depending on the strain rate) the mobility of these $\{112\} \langle 110 \rangle$ dislocations increases sufficiently for them to be regarded as glissile. The kink boundaries therefore then move under stress, and slip on the two 60° systems can interpenetrate.

Day and Stokes (1966a) later carried out similar tests but with a $[110]$ tensile axis which favors slip on oblique systems (i.e. at 120°). Two main modes of deformation were identified in this case:

(1) In the range 1400-1700°C, no interpenetration occurred and the crystals became subdivided into distinct blocks, slipping on different

systems, which were separated by kink boundaries. Brittle fracture occurred by void formation in the kink interfaces.

(2) Above $\sim 1700^{\circ}\text{C}$ (depending on the strain rate), slip on all systems interpenetrated and the crystals elongated about 100%, and recrystallized, before necking down to completely ductile fracture.

Stokes (1965a) also analyzed the temperature dependence of the flow stress, compensated by the change in shear modulus with temperature, from room temperature to 1000°C , and showed that the results exhibited two distinct regions:

(1) From room temperature $\sim 500^{\circ}\text{C}$, there was a strong, almost linear, decrease in flow stress with increasing temperature for as-received crystals. This decrease was greater, however, for crystals preannealed at 2000°C , suggesting that the high temperature anneal took some impurity particles into solution in the manner discussed previously.

(2) From 500 - 1000°C , the temperature dependence for the as-received crystals was identical to that of the shear modulus; but the curve for the preannealed crystals, when obtained by an increase rather than a decrease in temperature, showed a distinct hump with a peak at $\sim 800^{\circ}\text{C}$. It was suggested that this was probably due to re-precipitation. A similar temperature dependence for preannealed crystals has also been reported by May and Kronberg (1960) and Miles et al. (1966), and by Lewis (1966c) for vanadium-doped MgO. However, Miles (1964b) has pointed out that the hump in the curve observed by May and Kronberg (1960) was obtained by taking the maximum of the loading curve as the yield point; since this is followed by a sharp yield drop at temperatures greater than 700°C , a plot of the value of the stress after this yield drop largely

eliminates the hump.

The discussion so far has centered primarily around the results obtained from mechanical tests over a wide temperature range, and it is convenient at this stage to review briefly the observations reported using transmission electron microscopy. The first direct observation of dislocations in MgO using this technique, and in fact the first observation of dislocations in a refractory ceramic material, was by Washburn et al. (1960a, 1960b), in which thin foils were obtained by rotating the specimen above a jet of hot ortho-phosphoric acid. A modification of this technique was later presented by Kirkpatrick and Amelinckx (1962). In the study by Washburn et al. (1960a, 1960b), the motion of dislocations was observed by momentarily increasing the beam current between successive photographic exposures, and it was found that individual screw dislocations frequently cross-slipped from a (101) slip plane into a nearby parallel glide plane, and then back to the (101). This cross-slip leads to the formation of many highly elongated prismatic loops, which are observed profusely within slip bands produced by bending at room temperature but tend to break up into smaller loops when heated in the electron microscope. A mechanism was presented to explain the wide slip bands which are observed under the optical microscope (Washburn et al. 1959), by suggesting that two lengths of a screw dislocation may move apart by cross-slip until they are so far separated that they can each move independently on widely separated, but parallel, slip planes.

The elongated prismatic loops were later studied by Groves and Kelly (1961, 1962a), who showed that they became large circular loops after annealing at temperatures in the range 850-1600°C. It was

suggested that the process by which this occurred was the motion of vacant lattice sites along the dislocation lines and possibly through the bulk material. The loops so formed were of the vacancy type (Groves and Kelly 1962b), and lay on $\{110\}$ planes at 45° to the $\{100\}$ specimen plane, with a $\langle\bar{1}10\rangle$ Burgers vector. It was also shown (Groves and Kelly 1963a) that successive anneals increased the mean diameter of the loops and reduced their number; the mean loop diameter increased linearly with total annealing time within the limits 500-2000 \AA .

A study of as-received crystals showed that about half of the grown-in dislocations were contained in low-angle boundaries, while the rest tended to be present in small groups (Groves and Kelly 1963b). The immobility of grown-in dislocations, observed by Stokes (1962), could be explained since the dislocations in the low-angle boundaries did not generally lie on their slip planes, while the remaining dislocations had precipitates on them in the manner first reported by Venables (1961). Elkington et al. (1963) have also shown that the grown-in dislocations always contain precipitates along their length, and often do not lie in a slip plane, as shown in Fig. 17. However, a calculation revealed that the spacing of visible precipitates was not always sufficiently small to have pinned the dislocation at the maximum stress level reached in bending tests, suggesting that dislocations are immobile due to the presence of invisible impurity atmospheres or a high jog concentration. Precipitate particles along grown-in dislocations were identified as ZrO_2 particles by Venables (1963), and as $\text{MgO}\cdot\text{Al}_2\text{O}_3$ spinel particles by Henderson (1964). Groves and Fine (1964) studied an iron-doped MgO single crystal and reported the presence of MgFe_2O_4 precipitate. Stokes

(1966b) observed that particles of a MgMn_2O_4 phase precipitated preferentially on dislocation lines in a crystal into which manganese oxide had been diffused. The presence of precipitate particles along grown-in dislocations has also been studied in a number of other investigations (Bowen 1963, Bowen and Clarke 1963, Stokes and Li 1964b, Miles 1965).

It was reported by Groves and Kelly (1963b) that, while the dislocation structure of the slip bands was qualitatively similar in crystals deformed within the temperature range -196°C to 527°C , it appeared that the slip bands became somewhat wider and contained fewer dislocations as the temperature of deformation was increased. There was a change in dislocation structure in a crystal bent at 984°C , however, with long edge dislocations prominent and a marked reduction in the density of elongated loops and strings of circular loops. Elkington et al. (1963) also reported that the dislocation density continuously decreased with increasing temperature of deformation in the range -196°C to 1200°C , and noted further that there were only a few resolvable dislocation pairs in specimens deformed at -196°C although many were clearly visible at ambient temperature. The density of such pairs was found to decrease with increasing temperature, but the average pair size increased and the pairs broke into rows of small prismatic loops at temperatures of 750°C and above.

Stokes and Olsen (1963) suggested a possible mechanism whereby dislocation dipoles may be formed by the interaction of two screw dislocations of the same sign, when they became cross-linked to form an edge dislocation dipole at the cross-over point. Evidence for the occurrence of this mechanism in MgO was obtained by studying the dislocations

introduced into the foil after thinning. An alternative mechanism of dipole formation, whereby two edge dislocations of opposite sign approach each other and form a dipole along their line of contact, was considered unlikely for dislocations injected into thin foils after thinning, but may possibly account for many of the long dipoles seen in MgO after deforming in the bulk.

A detailed examination of the initiation of yielding in MgO was carried out by Stokes and Li (1964b), by introducing "fresh" dislocations by a prestrain at room temperature, and then aging these dislocations by a high temperature anneal. It was found that heat treatment above 600°C always resulted in an increase in the room temperature strength of the crystals due to dislocation locking. This locking was weak for temperatures in the range 700-1000°C, and yielding of the crystals occurred accompanied by a load drop and subsequent jerky flow. There was strong dislocation locking for heat treatments above 1000°C, however, and the specimens crushed in bending before slip was re-initiated in the tension surface after only a short anneal (e.g., 2 hours at 1200°C or 1/4 hour at 2000°C). It seems likely that this strong locking is due to the change in dislocation configuration which occurs at the higher temperatures (Stokes 1966b), since the much finer debris begins to anneal-out at temperatures as low as 700°C, and a series of closed prismatic loops are formed by annealing at 1000°C. These results, and those obtained later by Stokes (1966b), confirm the original observations of Groves and Kelly (1962a, 1963a). Specimens deformed at 1800°C were found to contain a cell structure with the cell boundaries consisting of stable dislocation networks of the type shown in Fig. 18.

While these results have referred to thin foils with the surface parallel to $\{100\}$, Ogawa (1966a, 1966b), Washburn and Cass (1966) and Cass and Washburn (1966a) developed methods for preparing thin foils parallel to the $\{110\}$ slip planes. Such foils are more advantageous than those parallel to the $\{001\}$ cleavage plane, since the $\{110\}$ slip planes do not lie at 45° or 90° to the surface and it is therefore possible to examine more than a thin strip of any given slip plane. Using this type of foil, Ogawa (1966b) showed that dipoles and slip dislocations were of mixed character, with no preference for edge or screw orientations. The occurrence of such effects as three-fold dipole nodes, observed also by Cass and Washburn (1966a), lead to the conclusion that these dipoles were produced by a collision mechanism, whereby a pair of dislocations move towards each other on parallel slip planes from two independent sources, rather than by the cross-slip of screw dislocations as suggested by Washburn et al. (1960a) and Groves and Kelly (1963b).

Cass and Washburn (1966a) and Washburn and Cass (1966) also noted that dipoles often deviated greatly from a pure edge orientation, although the shorter dipoles usually had a narrower spacing and lay closest to edge orientation. Both vacancy and interstitial dipoles were present in the deformed state, and, as with observations on foils parallel to $\{100\}$, the break up of dipoles into strings of prismatic loops was again observed at temperatures above 750°C . The dislocation structure in a crystal deformed at 850°C can be seen in Fig. 19, in which the black line, of $1\ \mu\text{m}$ total length, is parallel to both the active Burgers vector in the plane of the foil $\{110\}$ and the diffraction vector (after Washburn and Cass 1966). Many of the dipoles are transformed into

strings of loops by dislocation climb. While most crystals were deformed by loading in a $\langle 100 \rangle$ direction, some were also compressed in or near a $\langle 111 \rangle$ axis; in this case, the long dipoles were much less numerous and the short ones were often wide at the center and pointed at the ends. The results obtained from these investigations lent further support to a mechanism of dipole formation presented earlier by Washburn (1963).

These studies in transmission electron microscopy have, in general, attempted to directly correlate the observed dislocation configuration with the mechanical properties, but other experiments have also been conducted. For example, many measurements have been carried out of the mean and anomalous absorption coefficients of electrons in MgO (e.g. Kohra and Watanabe 1961, Watanabe et al. 1962, Kamiya 1963, Uyeda and Nonoyama 1965).

As work on MgO continues, it is becoming increasingly clear that alloying will be extremely important in the future, as it is with metals (see, for example, Clarke and Kelly 1963), and many further investigations are required to determine the mechanical properties of doped single crystals. Preliminary work by Satkiewicz (1961) indicated that unannealed crystals doped with Cr_2O_3 and NiO had higher yield stresses than the undoped material, and Liu et al. (1964) later showed that the addition of NiO produced up to a threefold increase in the compressive strength at room temperature. Day and Stokes (1967) continued this study to high temperatures, and found that with a 0.3% NiO additive, the alloys had lower yield stresses and lower ultimate tensile strengths compared with the pure material at all temperatures above 1200°C . It was suggested that this arose because the reducing atmosphere in the

furnace caused the alloy crystals to decompose by the removal of oxygen and internal precipitation of nickel, thus creating an excess of vacancies which enhanced the nonconservative mobility of dislocations. Thus it is concluded that a stable oxide such as MgO is strongly environment sensitive when an unstable oxide is present as an impurity. The addition of 1% of ferric iron to MgO was shown to roughly double the flow stress by Groves and Fine (1964), and the fracture toughness of MgO containing 0.1% titanium has been investigated by Groves and Shockey (1966). Other studies using doped crystals have also been reported by Ogawa (1966a, 1966b), in which electron microscopy revealed no appreciable difference in dislocation configuration between pure and doped crystals, Lewis (1966a, 1966b), Davidge (1967) and Matkin and Bowen (1965).

Many other studies using MgO single crystals have also been carried out: microstructure of cleaved surfaces (Lewis 1966b, Robins et al. 1966), effect of explosive shock (Gager et al. 1964, Klein 1965, Klein and Edington 1966, Klein and Rudman 1966), effect of neutron irradiation (Clarke and Sambell 1960, Bowen et al. 1962, Clarke et al. 1962a, Stablein 1963, Groves and Kelly 1963c, Desport and Smith 1964, Sambell and Bradley 1964, Bowen and Clarke 1964), variation of indentation hardness with temperature (Atkins et al. 1966), frictional behavior of crystals sliding over each other (Bowden et al. 1964, Bowden and Hanwell 1966, Bowden and Brookes 1966, Billinghamurst et al. 1966), effect of different chemical etches (Ghosh and Clarke 1961, Stiefbold et al. 1963, Cass and Washburn 1966b), effect of thermal shock (Miles and Clarke 1961, Clarke et al. 1961), and growth and strength of MgO whiskers (Hulse 1961, Wolff and Coskren 1965, Hulse and Tice 1965, Brubaker 1967). Other

studies have included measurements of internal friction (Dahlberg et al. 1962), the generation of vacancies by deformation (Klein and Gager 1966), and direct observations of the fracture of single crystals using high-speed photography (Field and Heyes 1965). Single crystals of MgO have also been studied using X-ray diffraction topography (Miuscov and Lang 1963, Lang and Miuscov 1964, Lang and Miles 1965, Lewis 1966b). Several investigators have studied the diffusion of impurities in MgO, and a recent tabulation of much of this work was provided by Harding (1967). Similarly, the major publications on the optical constants of MgO are listed in the recent report by Williams and Arakawa (1967).

III. BI-CRYSTALS

Bi-crystals are a convenient means of evaluating the basic mechanical behavior of a material since, unlike polycrystals, they allow a close study of such variables as the degree of misorientation across the boundary and the effect of the intersection of slip lines with the boundary plane. It should also be recognized that this configuration essentially corresponds to an "unconfined condition" and thus enables the study of the behavior of a boundary without the complications arising due to interactions between grains in a polycrystalline piece under stress. Furthermore, their use is particularly attractive in MgO, since they may be readily obtained by cutting directly from the large-grained magnesia blocks which are commercially available. As a result, many investigations have been reported using MgO bi-crystals (see Table I) and, while a number of problems are still unresolved, considerable progress has been made in interpreting the importance of grain boundaries during deformation.

In our present understanding of materials, the grain boundary may be considered to play two distinct, but inter-related, roles in determining the mechanical behavior. In the first place, the boundary may act as an obstacle to dislocation movement, and thus prevent the passage of slip lines from one grain to the next; in the second place, the boundary may directly contribute to the overall strain by the process of grain boundary sliding, whereby the two grains shear past each other along their mutual boundary. The phenomenon of grain boundary sliding is well documented in metals, but was not observed in a ceramic material until the bi-crystal experiments on sodium chloride and magnesia by Adams and Murray (1962). In compression tests on MgO bi-crystals containing asymmetrical boundaries, they found that sliding occurred at the boundary, and, within the limit of resolution of the optical microscope, the shear displacement was confined to the boundary plane. As with metals, sliding was observed to occur in discreet jumps, with the sliding rate dependent on stress and boundary misorientation. The possibility that sliding may have occurred merely by the shearing of a low strength, possibly glassy, boundary phase was discounted, since this would not have given rise to the observed orientation dependence. In later work, Murray et al. (1964) further examined the effect of stress and temperature, by testing bi-crystals with the boundary at 45° to the compressive axis. By conducting experiments in which the load was increased at regular intervals, they found that an incubation period always preceded the sliding and that this period decreased with increasing stress. The temperature was also found to be an important parameter in the range 1300-1500°C. This factor was later examined in more detail (Mountvala and Murray 1966) by testing

bi-crystals having boundaries of various misorientations. In this study, it was found that a critical temperature existed above which there was a catastrophic drop in strength; this critical temperature was $\sim 1200^{\circ}\text{C}$ for twist boundaries and $\sim 1400^{\circ}\text{C}$ for tilt boundaries, as shown in Fig. 20. In each case, fracture was intergranular, and caused by one grain sliding off the other. No convincing explanations were offered for this behavior. It may be significant that a brittle-ductile transition was observed by Hulse and Pask (1960) for single crystals of MgO tested in compression at a loading rate of 20 psi/sec in the $\langle 001 \rangle$ direction at 1200°C ; this behavior was presumably due to the development of easy intersections of conjugate slip planes at this temperature.

The effect of degree of misorientation has also been examined in other investigations. Johnston et al. (1962b) considered the case of a single slip band impinging upon the boundary, and showed that three separate situations may occur:

(1) For "small boundary misorientations (i.e., a simple tilt boundary) no cracks were formed, but the shear stress concentration ahead of the pile-up nucleated dislocation loops and therefore slip bands in the adjacent grain.

(2) For "medium" misorientations (i.e., a twist plus tilt boundary), the tensile stress concentration beneath an edge slip band nucleated a transgranular crack in the adjacent grain, and, under compression, this cleavage crack decelerated and generated dislocation loops which developed slip bands in the adjacent grain.

(3) For "large" misorientations (i.e., a large twist plus large tilt), the tensile stress concentration beneath an edge slip band

nucleated an intergranular crack.

Essentially similar results were reported by Sinha (1965), but in this case it was observed that micro-cracks were present prior to testing in the bi-crystals having large misorientations. Since these bi-crystals had been found to fracture intergranularly without any macroplasticity, it was suggested that the micro-cracks propagated at Griffith cracks along the boundary. While these results refer specifically to room temperature, tests at 1000°C revealed that the bi-crystals having a large misorientation then exhibited some plastic deformation prior to fracture, but failure again occurred by shearing along the boundary.

More recently, Lange and Buessem (1967) measured the intrinsic brittle strength for bi-crystals of various misorientations, where brittle fracture was defined as that occurring prior to any dislocation motion. In their tests, the specimens were chemically polished to remove all dislocation sources, and then a tensile load was applied and the specimens observed continuously under polarized light. By this procedure, cracks were found to nucleate at the boundary, and then either travel along the boundary completely (at low stress levels), or travel along the boundary and then deviate into one of the crystals (at high stress levels). In either case, a post-fracture examination always revealed secondary cracks in the crystals adjacent to the boundary, and it was suggested that intergranular fracture may predominate at the higher stress levels since the secondary cracks can then propagate more readily.

The first reported use of MgO bi-crystals to directly investigate the mechanism of fracture was that of Westwood (1961a, 1961b), wherein

tests were carried out to distinguish between the three types of failure which may occur in polycrystalline samples: by cracks initiating inside individual grains and then propagating transgranularly; by Zener-type cracks initiating at a boundary and propagating either intergranularly or transgranularly (Zener 1948); or by failure of the boundary itself due to impurity embrittlement or the presence of voids. The results confirmed the second of these alternatives, although in a somewhat modified form since, whereas the mechanism postulated by Zener (1948) for metals suggested that a crack may be formed at the boundary under the action of a piled-up group of edge dislocations, the results on MgO indicated that two such groups were required. A close examination of the bi-crystals led to the suggestion that cracks were only formed in the boundary when two slip bands, one from each grain, intersected the boundary separated by some small but finite ($\sim 1-2\mu\text{m}$) displacement. In particular, no cracks were observed associated with a single pile-up, and only occasionally when two slip bands met "head-on". This is shown in Fig. 21, where cracks have formed at points A and B but not at C, D, E or F. While this modification to the Zener mechanism is feasible at high strains, Westwood (1961a) suggested that at low strains it may occur if a source close to the boundary can be activated by the stress associated with a primary piled-up group of dislocations in the neighboring grain.

In later work, however, Johnson et al. (1962b) directly confirmed the classical Zener model by showing that cracks could be nucleated at a grain boundary by the pile-up of a single band of edge dislocations when bi-crystals were used in which fresh dislocation sources were eliminated by a high temperature anneal. It was therefore suggested

that the observation of Westwood (1961a) may represent a special case, wherein the small separation of 2 μm or less between the tips of the two slip bands is sufficiently small that the stress fields of the edge dislocations at the tips interact strongly. At larger separations, the interaction is much less, as the stresses fall off as the square of the separation distance, and cracks can no longer form by the superposition of the stress fields. Direct evidence for the Zener mechanism was also provided by Clarke et al. (1962a), by taking a ciné-film under polarized light conditions and observing the development of the stress birefringence patterns. However, it was also pointed out that the strain field due to the pile-up may have caused the growth of an existing micro-crack which was beyond the limits of microscopic resolution and possibly associated with impurity deposits (Venables 1961).

In a continuation of their single crystal work, Clarke et al. (1961) and Miles and Clarke (1961) studied the effect of thermal shock, under both very high (arc plasma) and moderate (quenching) rates of heat transfer. With the plasma discharge, they found that, while there were some instances in which cracks had clearly arisen at the boundary, it was often difficult to decide whether the point of origin was the grain boundary or cleavage steps near it. Since the boundaries were inefficient in preventing the passage of dislocations, it was again suggested that cracks at the boundaries may be due to pre-existing micro-cracks. The quenching tests confirmed that the boundary represents a source of weakness in the material.

Further direct confirmation of the Zener mechanism was provided by Ku and Johnston (1964) by measuring the crack nucleation stress, σ_F , a

function of the distance D between the source of the glide band responsible for crack formation and the grain boundary. To do this, bi-crystals were tested in four point bending, and fresh dislocation sources were introduced into the tension surface by means of micro-indentation. D was then taken as the distance measured along an edge slip band between the boundary and the point of indentation. The results showed that $\sigma_F = \sigma_O + KD^{-1/2}$, where σ_O is the stress required for dislocation multiplication, and K is a qualitative measure of boundary strength and thus of boundary porosity. It was therefore concluded that the stress concentration was primarily controlled by those dislocations of an appropriate sign emitted from the indentation towards the boundary, in confirmation of the Zener model.

A series of tests were carried out by Stokes and Li (1963b) in which bi-crystals were prepared with the boundary interface normal to the tensile axis, and with the surfaces chemically polished to eliminate all dislocation sources. Under these conditions the bi-crystals were capable of supporting very high stresses without fracture, with values approaching those obtained for polished single crystals. However, if the bi-crystals were sprinkled with silicon carbide, thereby introducing a high and uniform density of fresh dislocation sources, they were completely brittle and fractured at a stress level approximately equal to that required to initiate yielding in a sprinkled single crystal. A close study of the fracture surface showed that, while the fracture appeared almost completely intergranular, it had in fact originated from a cleavage crack nucleated by the direct inter-action of a single slip band with the grain boundary.

On the basis of their detailed observations, Stokes and Li (1963b) presented a fracture mechanism for bi-crystals tested under tension. The mechanism, as shown in Fig. 22, basically involves three stages.

(1) It commences when a single slip band in the grain AC intercepts the grain boundary along a line. Since the dislocations cannot cross the boundary, they are driven up against it by the applied stress, and the tensile stress concentration beneath the dislocation pile-up nucleates a transgranular cleavage crack lying in a (100) plane of the same grain.

(2) The crack is then opened up by the arrival of further dislocations, causing it to wedge deeper into grain AC. (3) At the same time, dislocations arriving at the bottom edge of the slip band open up an intergranular crack; finally, this crack propagates over the grain boundary surface. Thus, in tension, the cleavage crack is nucleated and propagates, into the grain which has slipped, in a manner consistent with experimental observations. In compression, however, the situation is reversed, and the cleavage crack is propagated into the adjacent grain, as shown in Fig. 23. Both of these mechanisms are consistent with the basic concept of the Zener model (Zener 1948), but it is not clear whether they support the results of Clarke et al. (1961) wherein it was suggested that the tensile stress concentration due to the pile-up acts on pre-existing submicroscopic flaws in the boundary. On the one hand, nucleation at such flaws satisfactorily explains the appearance of cleavage cracks at certain random points along the line of the slip band intersection with the boundary; it does not, however, explain the tendency for cracks to nucleate in the cleavage plane of the crystal rather than in the intergranular surface. Nevertheless, the results clearly show

that whenever slip occurs, a crack is nucleated at the boundary giving rise to immediate fracture. Thus the possibility of measurable plastic deformation in polycrystalline MgO under tension at room temperature appears extremely unlikely.

The role of impurities and defects in the grain boundary was studied by McPherson and Sinha (1965), by testing at room temperature bi-crystals and single crystals prepared from the same MgO block. Bi-crystals having a boundary with a simple, mostly tilt, misorientation were found to fail transgranularly after some plastic deformation, due to crack nucleation resulting from the pile-up of dislocations at the boundary. Bi-crystals with a large (both tilt and twist) misorientation, however, were found to fail intergranularly with no macroplasticity. In contrast, single crystals showed yielding and considerable plastic deformation. Since no slip was observed in the bi-crystals of large misorientation, it appeared that failure did not result from dislocation interaction; furthermore, very small microcracks could not be responsible since Clarke et al. (1962b) have shown that these do not act as Griffith cracks but grow as a result of preferential slip from their tips. The grain boundary was therefore examined in situ, and this revealed a series of cavities at the boundary, with a thickness of $\sim 0.1-1 \mu\text{m}$. An electron probe analysis of the fracture surface showed the existence of a silicate phase associated with the pores, and a microscopic examination revealed that failure occurred by cleavage between the cavities. This work therefore serves to emphasize the importance of defects in the boundary, since it was suggested that the cavities could act as a major source of weakness by propagating as Griffith cracks.

The tests described in this section refer specifically to bi-crystals. It is difficult to extend the data to the more complex case of polycrystalline materials. A first step in this direction was taken by Mountvala and Murray (1966) by testing tri-crystals in which each of the three grain boundaries had distinctly different misorientations. The results showed that the fracture behavior was controlled by the weaker boundary. It was thus suggested that in polycrystalline material, where boundaries of all misorientations are present, a marked loss in strength by deformation would be observed at temperatures of about 1200-1250°C when easy 90° intersections of slip systems becomes possible.

IV. POLYCRYSTALS

A. Production

A major difficulty which retarded the study of the mechanical properties of polycrystalline MgO for some years was the inability to obtain fully-dense material. Non-porous MgO is transparent; the presence of residual porosity causes opaqueness by scattering of light at the pores. The enormity of the task of completely removing all porosity can be best appreciated by pointing out that a porosity of 0.01 vol. % represents about 10^{11} pores, each of 0.1 μm diameter, per cubic centimeter. Polycrystalline ceramics are produced by sintering fine powders with or without pressure at elevated temperatures; invariably this results in small voids located at the grain boundaries causing opaqueness or, at best, translucency.

Although polycrystalline MgO of theoretical density is now available commercially (Eastman Kodak Company, Rochester, New York), the development is fairly recent. The first fabrication of optically transparent

MgO was by Rice (1962) who used a two-step process: hot-pressing MgO powder with a small additive of LiF, and a subsequent heat treatment. The suggested use of the lithium halides as a sintering aid arose since they allowed the functioning of a liquid phase sintering mechanism and their low boiling points provided the possibility of their being driven off to leave a dense, fine-grained material; ultimately, LiF was chosen as it appeared to give better results. The optimum density realized, with a 2% LiF additive, was 3.589 g/cc (Rice 1963), but this value must be accepted with reserve since it is slightly higher than the best value currently available for MgO single crystals (3.5833 ± 0.0010 g/cc, based on work by Skinner 1957). Budworth (1967) suggests the use of a value of 3.584 g/cc at room temperature for everyday ceramic purposes.

The hot-pressing step of the process was later studied by Benecke et al. (1967), in an attempt to determine the nature of the densification mechanism. An interesting sidelight of their investigation was that only certain sources of MgO powder produced transparent specimens; the reason for this is not fully understood, although a similar effect was also reported in the early work of Rice (1962). Miles et al. (1967) have pointed out that the starting powder should be of high purity, and it seems likely the relative surface area is of importance also. Using the apparently optimum additive of 2 wt % LiF, the favorable effect of LiF on the densification of MgO was clearly shown by Benecke et al. (1967) in the manner illustrated in Fig. 24. From curve B, it can be seen that with a constant heating rate densification begins at about 570°C, and is essentially complete by the time the melting point of LiF (846°C) is reached. In contrast, curve A shows that negligible densification

is attained in the absence of a LiF additive. Curve C indicates the importance of the nature of the powder, by showing that densification is delayed for mixtures containing the same MgO powder presintered at 1400°C for 5 hours. From this investigation, Benecke et al. (1967) showed that the hot-pressing step is controlled by a kinetic process; and it was pointed out that in order to develop transparency during the subsequent heat treatment, a theoretical density of at least 99.5% relative to MgO must be achieved during this first step. It was concluded that two densification mechanisms were capable of explaining the results: either a plastic deformation process, and/or an enhanced diffusional creep process at the grain boundaries.

A typical disc obtained by hot-pressing MgO powder with a 3% LiF additive is shown on the upper left in Fig. 25, (Langdon and Pask 1968a), together with a chemically-polished specimen of dimensions 0.25 x 0.25 x 1.0". In contrast, the opaque material on the upper right in Fig. 25 is sintered MgO obtained commercially. The microstructures of these two materials shown in the lower part of the figure indicate that although both have an average grain size of about 15-20 μm , the sintered material contains considerable porosity which is localized primarily along the grain boundaries and at the triple points. It should be noted that the measured density of the sintered material was of the order of 98.5% theoretical; the porosity is overemphasized in the figure due to the tendency for the pores to open up during the polishing procedure. Techniques for polishing polycrystalline MgO have been suggested by Gardner and Robinson (1962) and Copley and Pask (1965a).

The technique of Rice was further developed by Miles et al. (1967) so that fully transparent discs, of high optical perfection, were consistently produced. In this work, non-porous MgO was obtained for any additive of LiF in the range 0.1-5.0%, but it was reported that there was never more than a maximum of 0.5% LiF present in the compact after pressing; this was reduced to about 500 ppm by the subsequent heat treatment. The major factors affecting the perfection of the final product were found to be the homogeneity of mixing of the MgO and LiF, and the complete removal of gaseous contaminants by hot-pressing in vacuum. Since the process used by Miles et al (1967) for the production of fully-dense material has been well documented, it is summarized in detail in Table II.

Densification parameters for hot-pressing have recently been examined in detail by Rhodes et al. (1967), for both the technique of using the pure MgO powder and of using an LiF additive followed by an annealing treatment. It was found that the specimens were slightly more transparent when the additive was used (up to 85% total transmission in the visible spectrum for thin specimens produced with an additive compared with 60% total transmission without an additive). In addition, an anneal in a hydrogen atmosphere at 1250°C was found to be effective in improving the transparency of material produced without the additive (measured densities up to 99.9%), although considerable grain growth also took place (from 5 μm initially to 40 μm after 160 hours. By carrying out grain growth studies, weight loss experiments, and determinations of the lattice parameters, it was concluded that the densification mechanism in the presence of an additive was pressure-enhanced diffusion of the

Nabarro-Herring type (Nabarro 1948, Herring 1950). Since a calculation of the diffusion coefficient using this approach was of a reasonable magnitude, but between $\sim 10^7$ and $10^{9.5}$ faster than extrapolated values of the lattice diffusion coefficients, it was suggested that diffusion took place through a high-diffusivity grain boundary region or film. At higher temperatures, however, it was considered that a pressure-enhanced liquid phase sintering mechanism probably operated.

Additional tests were also carried out by Rhodes et al. (1967) to study the effectiveness of other lithium compounds as densification aids. These tests showed that:

(1) LiF was only effective as a densifying agent when it was present in concentrations of 0.5% or greater. (Miles et al. (1967) found they were able to produce transparent material with LiF additives as small as 0.1%, although in the complete absence of an additive the hot-pressing and post-sintering gave a material of only 70% of theoretical density.)

(2) Lithium oxalate (LiC_2O_4) was nearly as effective as LiF as a densifying agent, while LiOH and LiBr were quite ineffective. These results are summarized in Fig. 26, in which all pressings were conducted at a temperature of 800°C (with the exception of the 1% LiF additive for which the temperature was 750°C) under a pressure of 5000 psi. The curve for 0.25% LiF is similar to that obtained for pure MgO without an additive.

A more extensive study of the densification mechanisms involved in the fabrication of optically transparent polycrystalline specimens by hot pressing MgO powder with a LiF additive was undertaken by Hart et al. (1968). The densification progression was divided into three steps

with complex transition stages. The first step consists of a particle rearrangement process which is facilitated by the formation of a lubricating film on the particles. The second step consists of a redistribution of MgO by a solution-precipitation mechanism through a liquid phase consisting principally of LiF, but the controlling mechanism in the kinetic analysis is the flow of this liquid either within the compact or in being squeezed out of the compact. This step in other studies has been variously interpreted as diffusional creep along grain boundaries, pressure-enhanced diffusion of the Nabarro-Herring type, or grain boundary diffusion. The remaining LiF is lost in the third or annealing step by vaporization from the compact surface.

Spriggs et al. (1963) reported the production of a submicron grain size ($\sim 0.5 \mu\text{m}$) material, with a density greater than 99% theoretical, by a pressure consolidation technique utilizing a fairly low temperature (900°C) and high pressure (20,000 psi) with no additives. The measured tensile strength of this material ($\sim 15,000$ - $18,000$ psi) was of a similar magnitude to that obtained on the more usual 5-10 μm transparent specimens prepared by other methods; this relationship is inconsistent since there is evidence that strength decreases with increasing grain size (Spriggs and Vasilos 1963, Spriggs et al. 1962, Evans 1963). It was therefore suggested that the low value of tensile strength may be due to the presence of a small amount of residual porosity, but it may also be due to significant differences in the nature of the grain boundary. In later work, Spriggs et al. (1966) reported considerably higher strengths in bending of up to 28,600 psi and 48,800 psi for MgO pressure-sintered in air and in vacuum, respectively. By comparison, the equivalent value

obtained for the commercially available transparent MgO used for infrared transmitting elements is about 35,000 psi.

A study of grain growth in fully-dense material of this type (Spriggs et al. 1964) showed good agreement with the theoretical relationship for normal isothermal grain growth, although the observed activation energy of about 81 kcal/mole is greater than the value of 60 kcal/mole obtained by Daniels et al. (1962) for porous MgO. Rice (1966a) has demonstrated the ability of voids to inhibit grain growth, and this is substantiated here since the growth rates observed by Spriggs et al. (1964) were very much faster than those recorded by Daniels et al. (1962).

Using pressure sintering, Vasilos and Spriggs (1963, 1965) were able to obtain optically transparent MgO without the use of a liquid-phase (LiF) additive, and, by taking porosity into account in calculating diffusion coefficients, it was concluded that the densification in this process was essentially diffusion-controlled. This technique has also been used for the production of other ceramics such as Al_2O_3 and NiO (Vasilos and Spriggs 1965, Spriggs 1965). These investigations refuted the possibility of plastic flow as the predominant mechanism due to (1) the non-existence of a true end-point density and (2) the absence of extensive preferred orientation. It may be necessary to modify this statement, however, since Tagai et al. (1967) have recently reported the existence of a high degree of preferred orientation in hot-pressed specimens, with the (111) plane predominantly oriented in a direction perpendicular to that of the applied pressure. Furthermore, this preferred orientation is not eliminated by annealing and considerable consequent grain growth. In contrast, de Wys and Leipold (1967) reported that no

preferred orientation was present in a hot-pressed material with a grain size of 5 μm , produced by the technique of Neilsen and Leipold (1963), but that a preferred orientation developed on subsequent heating due to grain growth. It was suggested that this condition was probably due to the presence of a thermal gradient.

Stokes and Li (1963b) have shown that specimens produced in the manner of Vasilos and Spriggs (1965), with a density of 3.581 g/cc and no visible porosity after hot-pressing, have a second phase present along the triple lines where the grains intersect. This phase disappears during annealing at 2000°C, probably by diffusion along the triple lines and subsequent evaporation at the surface, resulting in slight porosity at the intergranular surfaces. Rice (1966a) has also reported the presence of a second phase in material of this type.

Other methods for producing polycrystalline MgO are also available. Day and Stokes (1966a, 1966b, 1966c) have shown that single crystals with a $\langle 110 \rangle$ tensile axis recrystallize when pulled in tension to high strains (60%) at 1800°C, producing polycrystalline specimens with a fine grain size. A short anneal at 2100°C produces a material that is fully-dense and completely free from any pores or flaws, with an average grain size of about 1000 μm . Harrison (1964) has produced material of up to 99.5% of theoretical density by an isostatic pressing and sintering technique, but it has been pointed out (Stokes and Li 1963b) that sintered material invariably contains some porosity and is therefore unlikely to achieve the same high tensile strengths as hot-pressed material.

It is also possible to produce polycrystalline MgO by hot extrusion, in the manner developed by Rice and Hunt (1966, 1967). In this technique MgO and various MgO alloys, in the form of solid billets obtained by sintering or fusion, were extruded in tungsten cans; densities of up to ~ 3.60 g/cc were achieved. All extruded MgO bodies had a strong $\langle 100 \rangle$ texture parallel to the extrusion axis, and this was found to give an increase in strength of 50-100% over hot-pressed material of equivalent grain size provided the tensile stress was parallel or in one of two directions perpendicular to the extrusion axis. In the other perpendicular direction, the strengths of the two materials were comparable. It was also observed that there was a weakening of the unannealed body if chemical polishing was carried out, which, it was suggested, was due to a stress corrosion mechanism.

Since the major interest has been the production of fully-dense MgO, only limited work has been carried out on the possibility of irregular impurity distribution within the final material. Leipold (1966) has shown that in high-purity hot-pressed material some common impurities, such as Al, Ca and Si, are segregated at the grain boundaries, even when only present in amounts as small as 30 ppm. Later work (Leipold 1967) established this effect to be primarily due to the slow cooling employed after hot-pressing, although it could not be fully eliminated even by a rapid quench. Such segregation would be expected to have a marked effect on the mechanical properties of the material, particularly those involving grain boundary processes. For example, Nicholson (1966) studied grain growth in MgO with a 1% Ti (as TiO_2) or a 1% Fe (as Fe_2O_3) additive, and found that the activation energies for grain growth were $10^4 \pm 20$ and

146 \pm 25 kcal/mole, respectively. These values are much higher than the activation energy of 81 kcal/mole obtained by Spriggs et al. (1964) for pure MgO.

B. Mechanical Properties

The effort necessarily expended in developing techniques for the production of fully-dense polycrystalline MgO and the indicated uncertainties in the characterization of resulting specimens have done much to inhibit investigations of the mechanical properties of the material in other than single crystal form. Notwithstanding this, however, detailed investigations have been carried out to compare the behavior of different types of hot-pressed material with that of recrystallized single crystals (Day and Stokes 1966b, 1966c), the behavior of materials produced by different techniques (Copley and Pask 1965a) and, more recently, the differing behavior of materials with various porosities (Langdon and Pask, 1968b).

In order for a polycrystalline body to deform plastically, without the nucleation of voids at the internal boundaries, each of the individual grains must be capable of a perfectly general change in shape. This leads to the von Mises criterion (von Mises 1928), which states that, for polycrystalline plasticity, the solid must possess five independent slip systems (an independent slip system is one capable of producing deformation which cannot be accomplished by a linear combination of slip on the other available systems). In the case of MgO, Groves and Kelly (1963c) have shown that slip in $\langle 110 \rangle$ directions over $\{110\}$ planes as observed in single crystals at low temperatures, gives only two independent slip systems. However, slip in $\langle 110 \rangle$ directions

over {100} planes corresponds to three independent slip systems, so that a combination of both {110} and {100} slip, provided they operate independently, satisfies the von Mises criterion. The onset of fairly easy slip on {100} planes above about 1500°C, as indicated in single crystal experiments, should then give rise to a transition from brittle to ductile behavior. Any movement that may materialize on {111} planes which would provide five independent systems, would contribute to this transition. In fact, this analysis oversimplifies the problem since slip is not homogeneous, as assumed in the von Mises criterion. Slip is confined to discrete slip bands, and it may also be difficult, as shown for single crystals, for the various slip systems to interpenetrate each other. Furthermore, at high temperatures, greater than about $0.5 T_m$, other factors contribute to polycrystalline plasticity, such as polygonization of dislocations in highly strained regions, grain boundary migration which may act as a stress relief mechanism, and grain boundary sliding. These factors lead to complexities in interpreting the results of any mechanical tests on polycrystalline MgO at elevated temperatures.

Day and Stokes (1966b, 1966c) compared the mechanical behavior of materials produced by recrystallization with that of material produced by hot-pressing both with and without a LiF additive. In this study, all of the specimens were given the same annealing treatment (1 hour at 2000°C) to standardize the initial condition. Specimens were tested in tension, at a strain rate of $\sim 5 \times 10^{-4} \text{ sec}^{-1}$, and it was found that the recrystallized material exhibited a brittle-ductile transition at a temperature of about 1700°C. This is evident from a comparison of the stress-strain curves obtained at various temperatures, as shown in Fig. 27.

At temperatures of 1600°C and below, the behavior was completely brittle, although optical examination revealed that a limited amount of slip had taken place primarily in the vicinity of triple points. At 1800°C and above, recrystallization occurred at very large strains (~80% elongation) and the specimens necked down to a ductile fracture after up to 100% homogeneous elongation. An examination of the specimens deformed at 1700°C showed considerable evidence of grain boundary migration, grain boundary sliding, and the formation of substructure within the grains due to dislocation polygonization. Brittle intergranular fracture took place at this temperature, which, it was suggested, was due to internal voids produced on the boundaries by the grain boundary sliding. Such sliding is known to be possible at these temperatures, as shown by the experiments of Adams and Murray (1962) and Copley and Pask (1965a), and will lead to void formation and consequent brittle fracture if the temperature is sufficiently low that the individual grains are still not plastic enough to follow the flow patterns required to maintain the integrity of the matrix. Thus the brittle-ductile transition is not able to take place when the additional slip systems become available at 1500°C, and the transition is delayed until about 1700°C.

With the hot-pressed material, it was observed that specimens containing porosity were in general weaker than those relatively free from pores, when the grain size was comparable. However, at temperatures below 2000°C, a fine-grained material containing a slight amount of residual porosity was, in fact, superior to a pore-free material having a large grain size; this situation was reversed at temperatures greater than 2000°C. All of these specimens exhibited brittle behavior up to

about 1800°C, but elongations of the order of 30% were achieved at 1200°C; there was evidence of the existence of a brittle-ductile transition at temperatures somewhat higher than this. Fracture surfaces were always completely intergranular above 1800°C, but showed considerable evidence of cleavage at lower temperatures. Surface folds were often observed spreading out from the triple points in specimens deformed to fracture (21% total elongation) at 1900°C, as shown in Fig. 28. No definitive reason could be given for the observation that it was necessary to go about 500°C higher in temperature with the hot-pressed material to observe the same phenomena as with the recrystallized specimens, although it was suggested that such factors as residual porosity and imperfections would play an important role. The over-riding conclusion from this work, however, is the close similarity between the high temperature deformation processes in polycrystalline MgO and those already documented in some detail for the face centered cubic metals. Since the primary slip parameters of the f.c.c. metals are $\{111\} \langle 1\bar{1}0 \rangle$ and since slip can also occur on $\{100\}$ and $\{110\}$ planes, this similarity is not too surprising.

In a manner similar to that reported for single crystals, Stokes and Li (1963b) have shown that there is a substantial drop in the tensile strength (by a factor of about one third) for polycrystalline specimens sprinkled with silicon carbide powder. This is almost certainly due to the introduction by sprinkling of fresh mobile dislocations although, unlike single crystals, an attempt to reveal evidence of this by etching was unsuccessful. Thus, even with polycrystals, the extreme sensitivity to surface conditions is demonstrated, and high tensile strengths are best attained by using fully-dense chemically-polished material. The

advantage of chemical polishing with slightly porous material has been amply demonstrated by Harrison (1964).

To check the behavior of materials produced by different techniques, Copley and Pask (1965a) obtained stress-strain curves for five types of polycrystalline MgO, all nominally dense and pure but differing in grain size, composition and porosity. Tests were conducted in compression at temperatures up to 1500°C, under a constant initial force rate of 20 psi/sec. The results revealed marked differences in behavior. One material, produced by isostatic pressing and sintering, showed substantial ductility at temperatures as low as 800°C, and the other materials only showed ductility at temperatures of about 1200°C and above. The differing behavior is best appreciated by comparing the observed yield stresses at different temperatures with those obtained for single crystals (Hulse et al. 1963, Copley and Pask 1965a), as shown in Fig. 29, where the yield stress values for single crystals with $\langle 100 \rangle$ and $\langle 111 \rangle$ stress axes are plotted as a function of temperature. The "type 1" material, which was produced by hot-pressing with an LiF addition, only first showed ductility at about 1200°C, whereas the "type 2" material formed by sintering was one that exhibited ductility at temperatures down to less than 800°C.

It is clear that the "type 2" specimens yielded at stresses which were considerably lower than those needed to yield a single crystal with a $\langle 111 \rangle$ stress axis, although the "type 1" specimens showed approximate agreement with the $\langle 111 \rangle$ data for single crystals. The yielding behavior of the "type 2" specimens can be quantitatively explained by noting that slip first occurs on the $\{110\} \langle \bar{1}\bar{1}0 \rangle$ slip systems causing the build-up of long range stresses within the grains. Such stresses can then force

slip on the $\{001\} \langle 1\bar{1}0 \rangle$ slip systems at temperatures above the brittle-ductile transition, but will lead to grain boundary separation below the transition temperature. It follows therefore that the temperature of transition will depend not only on the mobilities of dislocations on these two slip systems but also on the strength of the grain boundaries. A possible explanation of the behavior of the "type 2" material is that the few pores present, which do not occur in the "type 1" specimens, are sufficient to initiate slip and develop ductility. The less ductile behavior of the "type 1" specimens may also be due to the presence of Li and F as a result of the hot-pressing, which would possibly reduce the mobility of dislocations (see, for example, the single crystal data of Gorum et al. 1960); or, if present predominantly as a thin second phase or in a zone along the grain boundaries, may severely limit ductility by permitting the relief of stresses between grains by localized boundary sliding. Direct evidence for the existence of sliding at the higher temperatures was provided by the loss of transparency which took place during the test, and by the offsets of surface marker lines. An example of such offsets is shown in Fig. 30 for a hot-pressed specimen strained 3% at 1400°C.

An attempt was made using transmission electron microscopy to determine whether there is in fact a thin second phase present at the grain boundaries of material hot-pressed with a 3% LiF additive and heat treated. Thin foils for electron microscopy can be obtained by a fracture method (Hulse and Tice 1966), but this has the disadvantage of yielding only extremely small foils which rarely contain even low-angle grain boundaries. A chemical thinning technique was therefore developed

(Langdon 1967) although this is not easily applied to porous material since the pores are subject to preferential attack. The technique can, however, be used with fully-dense MgO, but care must be taken to avoid introducing mechanical damage during the handling of the foil; an example of such damage is shown in Fig. 31 where dislocations have been introduced into the dislocation-free grain by cutting of the foil at point A. On examination, neither the deformed nor undeformed specimens showed any evidence of a grain boundary second phase in any of the many foils studied (Langdon and Pask 1968a). In contrast, Rhodes et al. (1967) have recently reported the existence of a grain boundary film immediately following the hot-pressing state in material hot-pressed with a 2% LiF additive.

Relatively few dislocations were observed in specimens deformed in compression at temperatures less than 1200°C (Langdon and Pask 1968a), but at higher temperatures, when slip took place on both the $\{110\} \langle 1\bar{1}0 \rangle$ and $\{001\} \langle 1\bar{1}0 \rangle$ slip systems and 90° intersection of the $\{110\} \langle 1\bar{1}0 \rangle$ systems was also possible, many dislocations were visible within the grains and specimens fractured at total strains in excess of 10%. At these temperatures, bulk diffusion occurs and the dislocations are able to climb out of their slip bands to annihilate or polygonize.

The effect of varying porosity on the deformation of polycrystalline MgO has been investigated by testing specimens of varying density and with pores located primarily either along the grain boundaries or within the grains (Langdon and Pask, 1968b). The results provide conclusive proof that for specimens of similar density, the stress-strain behavior is critically dependent on the location of the pores. With pores located

intergranularly, or for specimens of theoretical density, ductility only occurs at temperatures greater than about 1200°C; whereas specimens containing transgranular pores show ductility at temperatures as low as 800°C, in the manner first reported for the "type 2" specimens of Copley and Pask (1965a). It is concluded therefore that the pores play an important role in the polycrystalline behavior, and that ductility is best realized if the pores are not present on the grain boundaries, or if present, are very small relative to the average grain size.

It is interesting to note that brittleness was always observed in the tensile tests at temperatures as high as 1600°C (Day and Stokes 1966b, 1966c), whereas the investigations in compression revealed appreciable ductility at temperatures as low as 800°C. This apparent dichotomy is probably due to the onset of grain boundary sliding, which may give rise to extensive void formation in tension and therefore lead to brittle fracture.

The results so far described have been essentially exploratory in nature, due to the present difficulties of producing polycrystalline material with controlled microstructures. Thus, while these tests give an indication of the type of behavior to be expected from polycrystalline MgO produced by various techniques, they are only a first step towards an overall understanding of the mechanisms of plastic deformation of nominally single phase specimens. Other types of investigation have also been carried out; for example, Davidge and Tappin (1967) studied the effect of thermal shock on fully-dense MgO by quenching heated specimens in water; Spriggs and Vasilos (1963), Vasilos et al. (1964b) and Jacobson and Fehrenbacher (1966) measured the transverse bend strength with particular

reference to the effect of grain size and porosity; Rice (1966b) determined the fracture strengths of hot-pressed and hot extruded MgO; and Tattersall and Tappin (1966) measured the "work of fracture" in MgO polycrystals (i.e., the energy absorbed as a crack grows -- see also Clarke et al. 1966). These latter results are particularly interesting since they show that a theoretically dense material has a lower work of fracture than a porous material, thereby indicating that the presence of pores gives rise to an energy-absorbing process. Values of the elastic modulus have also been measured, both at 25°C (Chung 1963) and as a function of grain size and temperature (Vasilos et al. 1964b), and single crystal values have been used to calculate the elastic constants of the polycrystalline material (Lowrie 1963). Recently, measurements of the elastic moduli have also been extended to MgO-pore and MgO-nickel-pore composites (Janowski and Rossi 1967), and the results show that the inclusion of pores or nickel particles produces a decrease in the elastic moduli of the composite which is greater than that predicted by theory. It is therefore clear that measurements of this type will become increasingly important in the future, particularly when a better control over the degree of porosity, and thus of microstructure, is attained.

V. CREEP

"Creep" may be defined as the time-dependent plastic flow which occurs when a constant load or stress is applied to a material over a prolonged period of time. This flow is a thermally activated process, and is of particular importance at temperatures greater than about $0.5 T_m$. If i atomistic mechanisms are contributing to this plastic deformation, then the creep rate, $\dot{\epsilon}$, can be represented by an equation of the form

$$\dot{\epsilon} = \sum_i A_i f_i (\sigma, T, S) \exp [- Q_i (\sigma, T, S)]/RT \quad (3)$$

where A is a constant, f_i represents the functional dependence for the i^{th} process, σ is the applied stress, T is the absolute temperature, Q is the activation energy for creep, R is the gas constant, and S is a structure parameter which varies with grain size, substructure and porosity.

In practice, one creep process is usually dominant, and the creep rate is often then described by an equation of the form

$$\dot{\epsilon} = A' \sigma^n \exp(-Q/RT) \quad (4)$$

where n is the stress exponent and A' is approximately constant for any given stress (the approximation arises due to a slight dependence on temperature).

It is known that a number of processes may be involved in the deformation of materials at high temperatures, such as the glide, cross-slip and climb of dislocations, grain boundary sliding, and vacancy diffusion; and the problem therefore arises of determining which mechanism is rate-controlling under any specified conditions. This determination is best achieved by measuring the activation energy, Q , and stress exponent, n , from tests covering a wide range of temperature and stress. The stress exponent is particularly important in distinguishing between various rate-controlling mechanisms, since the stress-directed diffusion of vacancies requires that $n = 1$, whether diffusion is through the lattice

(Nabarro 1948, Herring 1950) or along the grain boundaries (Coble 1963), whereas models based on the movement of dislocations through the lattice require $n = 3$ or greater (Weertman 1955, 1957, Chang 1963).

While the creep of metals is fairly well documented, this is unfortunately not the case for ionic materials. A comprehensive summary of the results obtained from creep tests on single crystal and polycrystalline MgO is given in Table III, but it is not easy to draw definitive conclusions from this data. Firstly, the majority of tests have been conducted in bending, wherein an interpretation of the results is difficult since both the stress and strain rate vary throughout the cross-section of the specimen. This problem is overcome to a large degree in the compression tests, but difficulties then arise due to the possibility of plastic buckling. Such buckling was analyzed in detail by Cropper (1966), and it was shown that the compressive load at which plastically deforming MgO becomes unstable is accurately predicted from a consideration of continuum mechanics. It was also shown that buckling may be eliminated for most test conditions by the application of suitable end constraints and a careful choice of specimen geometry. Secondly, problems may arise due to the presence of voids, particularly if these are preferentially located at the grain boundaries and thereby increase the ease of grain boundary sliding and hinder migration, or due to the segregation of impurities to the grain boundaries. Leipold (1966) has shown that segregation can occur even when the impurities are present in low concentrations (~ 30 ppm); this can substantially change the grain boundary diffusion coefficients (Wuensch and Vasilos 1966), and thus alter the mechanical behavior of the material.

Without disregarding these limitations, however, a quantitative assessment of the tabulated data is possible. For single crystals, there is an obvious discrepancy between the high temperature results of Rothwell and Neiman (1965) where $n = 3$ independent of temperature, and those of Cummerow (1963) where $n = 4 - 7$ with a tendency for the exponent to increase with decreasing temperature. The activation energy obtained by Rothwell and Neiman (1965) at the higher temperatures is in fair agreement with the value of 120 kcal/mole reported by Davies (1963) for pair formation and diffusion of oxygen in MgO, and it was therefore concluded that creep is oxygen ion diffusion-controlled by the climb of edge dislocations over obstacles. However, it was suggested that at 1000°C ($\sim 0.4 T_m$), where the activation energy is considerably lower, creep is controlled by the cross-slip of screw dislocations. The difference in behavior between 1000°C and 1600°C is clearly indicated by the etch-pit patterns obtained after creep at these two temperatures, as shown in Fig. 32; discrete slip bands are visible at the lower temperature (top photo), but there is a polygonized structure at 1600°C (bottom photo).

Working in compression, Cropper (1966) obtained a stress exponent of 4.7 at 1200°C, which is intermediate between the values obtained by Rothwell and Neiman (1965) at 1000°C and 1300-1630°C, respectively. This value was interpreted in terms of a model based on the dissolution of trails left behind by moving screw dislocations (Chang 1963), in which n is 5 or 4 for the dissolution of dipoles or loops, respectively. Some support for this suggestion is provided by the observation that the motion of dislocations in MgO results in dislocation dipoles at low temperatures, and these tend to break up into prismatic loops at temperatures greater

than about 800°C (Groves and Kelly 1962a, Stokes 1966b, Washburn and Cass 1966, Cass and Washburn 1966a).

For the creep of polycrystals, the situation is more complex. Testing in bending Vasilos et al. (1964a) obtained a linear stress dependence for very small grain sizes (1-3 μ m), which implies a stress-directed diffusional mechanism. A linear stress dependence was also reported by Passmore et al. (1966) for grain sizes of 5.5-20 μ m. Testing in compression, however, Langdon and Pask (1968b) obtained an exponent of 3.3 for grain sizes in the range 12-52 μ m, thereby suggesting a dislocation mechanism such as the climb of edge dislocations over obstacles.

The activation energy for creep obtained by Vasilos et al. (1964a) is intermediate between the values of 79 kcal/mole (Lindner and Parfitt 1957) and 62.4 kcal/mole (Oishi and Kingery 1960) for lattice self-diffusion in MgO of the cation and anion respectively; although, since the anion is the slower moving species, it is anticipated that it should be rate-controlling. The value of 54.1 kcal/mole obtained by Passmore et al. (1966) shows fair agreement with that for extrinsic oxygen ion diffusion, and their results also revealed that the strain rate was inversely proportional to $d^{2.5}$, where d is the average grain size, in the range 5.5-20 μ m. This grain size dependence is intermediate between the d^2 predicted for lattice diffusion (Nabarro 1948, Herring 1950) and the d^3 predicted for grain boundary diffusion (Coble 1963); and, after a consideration of the calculated diffusion coefficients, lead to the proposition of a vacancy formation mechanism. In the work of Langdon and Pask (1968b), however, no grain size dependence was observed in the range 12-52 μ m. The larger values for the activation energy obtained by

Passmore et al. (1966) for grain sizes less than 5.5 μm , indicate a possible transition to control by intrinsic magnesium ion diffusion; although, since the oxygen ion is the slower moving, it is not clear why this should occur.

It is well known that in metals the grain boundaries play an important role in the creep behavior at elevated temperatures. In ionic materials complexities arise due to the possibility of impurity segregation at the boundary, the presence of voids, and the nature of the chemical bonding at the grain boundary. Thus, the boundaries cannot be thought of simply in terms of the random misfit of two grains, or in terms of a specific dislocation model, as applied to metals, due to the possibility of large local lattice strains, charge faulting, and composition changes. These problems have been discussed in some detail by Rice (1966a) and Carniglia (1966); and Smyth and Leipold (1967) have recently developed a model for grain boundaries in MgO in terms of a random network.

The occurrence of grain boundary sliding during creep at high temperatures is known to make a substantial contribution to the overall strain of metals, particularly under conditions of small grain size and low strain rate, and it is therefore probably of importance in ionic polycrystals also (see, for example, Habraken and Blavier 1963, Hornstra 1965). Hensler and Cullen (1967) attempted to determine the grain boundary sliding contribution, in polycrystalline MgO of 98% theoretical density, by measuring the change in grain shape which occurred during compressive creep. Their results suggested that sliding accounted for all of the elongation at temperatures in the range 1200-1500°C, up to 30% total

strain; this conclusion, however, must be accepted with reserve since experiments on metals have shown that the grain shape technique often leads to an overestimation of the grain boundary sliding contribution due to the tendency for migration to spheroidize the grains (Langdon and Bell, 1967). If spheroidization is unimportant in MgO, which has yet to be proven, the lack of any observable change in grain shape in the experiments of Hensler and Cullen (1967) appears to negate the possibility of a stress-directed diffusional mechanism, either through the lattice or along the grain boundaries, since both models predict a change in grain shape corresponding to strains of the same magnitude as the total specimen strain. Gifkins (1968) has recently developed a model of creep in which grain boundary protrusions move along the boundary by diffusion-controlled sliding and are accommodated by diffusion around triple points. This mechanism, which yields a linear stress dependence, does not require a permanent change in grain shape, and would appear to satisfy the results of Hensler and Cullen (1967).

Other factors affecting the creep of MgO have also been investigated. It has been shown, for example, that the application of an electric field to a single crystal during creep markedly affects the movement of charged dislocations and leads to an enhanced steady-state creep rate (Neiman and Rothwell 1963, Rothwell and Neiman 1965, Rothwell 1966). The effect on the creep rate of porosity (Spriggs and Vasilos 1964) and the minor addition of various oxides such as CaO and ZrO₂ (Vasilos et al. 1965) has also been examined. Further investigations of this type are necessary before the creep processes in MgO are fully understood.

VI. CONCLUSIONS

It is evident that the level of understanding of the mechanical behavior of single crystals of MgO is considerably advanced. Many studies in the last ten years have contributed significantly to a knowledge of dislocation motion and interactions, and crack nucleation and growth. This development was due to the easy availability of single crystals and the application of the electron microscope to single crystal studies. By the same token, the effect of impurities and alloying on the mechanical behavior of MgO single crystals has not been sufficiently studied, in this case as a result of the lack of such controlled specimens. It is expected that this area will receive more attention in the near future because of the accompanying potential beneficial effects of reducing the tendencies for cleavage fracture. It is a paradox that the factor of easy cleavage, which was responsible for initiating ductility studies in ionic crystals, by making it possible to obtain suitable specimens free of Griffith cracks in the surface, is also the characteristic that is probably most responsible for brittle behavior at low temperatures.

The single crystal and bicrystal studies provide a good background for investigations of the mechanical behavior of polycrystalline specimens. The latter studies, however, have been limited primarily because of a lack of capability of fabricating specimens with controlled microstructure, or character, and also of characterizing them. It is difficult to carry on fundamental studies based on relating character to mechanical behavior when, for example, the nature of the grain boundary or the distribution of impurities are not too well known. Nevertheless,

a potential interest exists for following up these factors whenever the opportunity arises in the form of adequate specimens, as evidenced by the reported studies on polycrystalline and creep behavior.

The current interest in developing a science of ceramic processing, and the progress that has already been made in producing theoretically dense polycrystalline pieces, indicates that specimens with controlled character should be available in the not-too-distant future. Another important contributing factor to propagating polycrystalline studies is the availability of relatively new analytical tools--the electron microprobe and the electron scanning microscope.

Demands for materials with high temperature capability are increasing rather than decreasing. Therefore, regardless of whether materials with ductility are developed or not, detailed fundamental studies on the mechanical behavior of MgO and other ceramic materials primarily in terms of nucleation and propagation of fracture, and relating them to the material character, will continue.

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REFERENCES

- Adams, M. A., and Murray, G. T. (1962). J. Appl. Phys., 33, 2126.
- Alden, T. H. (1963a). Appl. Phys. Letters, 2, 107.
- Alden, T. H. (1963b). Trans. AIME, 227, 1103.
- Argon, A. S. and Orowan, E (1964a). Phil. Mag., 9, 1003.
- Argon, A. S. and Orowan, E. (1964b). Phil. Mag., 9, 1023.
- Atkins, A. G., Silvério, A., and Tabor, D. (1966). J. Inst. Met., 94, 369.
- Atkins, A. G., and Tabor, D. (1967). J. Am. Ceram. Soc., 50, 195.
- Bakunov, V. S., Lukin, E. S., and Poluboyarinov, D. N. (1966). Tr. Mosk. Khim-Tekhnol. Inst., (50), 216.
- Benecke, M. W., Olson, N. E., and Pask, J. A. (1967). J. Am. Ceram. Soc. 50, 365.
- Billinghamurst, P. R., Brookes, C. A., and Tabor, D. (1966). Proc. Conf. on the Physical Basis of Yield and Fracture, Oxford, Inst. of Physics and Physical Soc. (London), pp. 253-258.
- Bowden, F. P. and Brookes, C. A. (1966). Proc. Roy. Soc., 295A, 244.
- Bowden, F. P. and Hanwell, A. E. (1966). Proc. Roy. Soc., 295A, 233.

- Bowden, F. P., Brookes, C. A., and Hanwell, A. E. (1964). Nature, 203, 27.
- Bowen, D. H. (1963). Trans. Brit. Ceram. Soc., 62, 771.
- Bowen, D. H. and Clarke, F. J. P. (1963), Phil. Mag., 8, 1257.
- Bowen, D. H. and Clarke, F. J. P. (1964). Phil. Mag., 9, 413.
- Bowen, D. H., Wilks, R. S. and Clarke, F. J. P. (1962). J. Nucl. Materials,
6, 148.
- Briggs, A. and Clarke, F. J. P. (1965). Proc. First International Conf.
on Fracture, Sendai, Japan, 2, 1321.
- Briggs, A., Clarke, F. J. P., and Tattersall, H. G. (1964). Phil. Mag., 9
1041.
- Brubaker, B. D. (1967). J. Am. Ceram. Soc., 50, 624.
- Bruneau, A. A. and Pratt, P. L. (1962). Phil. Mag., 7, 1871.
- Budworth, D. W. (1967). J. Brit. Ceram. Soc., 4, 481.
- Carniglia, S. C. (1966), Materials Sci. Res., 3, 425.
- Cass, T. R. and Washburn, J. (1966a). Proc. Brit. Ceram. Soc., (6), 239.
- Cass, T. R. and Washburn, J. (1966b). J. Am. Ceram. Soc., 49, 343.
- Chang, R. (1963). In "The Physics and Chemistry of Ceramics" (C. Klingsberg,
ed.), pp. 275-285, Gordon and Breach, New York.

Chung, D. H. (1963). Phil. Mag., 8, 833.

Clarke, F. J. P. and Kelly A. (1963). Trans. Brit. Ceram. Soc., 62, 785.

Clarke, F. J. P. and Sambell, R. A. J. (1960). Phil. Mag., 5, 697.

Clarke, F. J. P., Sambell, R. A. J., and Miles, G. D. (1961). Trans. Brit. Ceram. Soc., 60, 299.

Clarke, F. J. P., Sambell, R. A. J., and Tattersall, H. G., (1962a). Trans. Brit. Ceram. Soc., 61, 61.

Clarke, F. J. P., Sambell, R. A. J., and Tattersall, H. G. (1962b). Phil. Mag., 7, 393.

Clarke, F. J. P., Tattersall, H. G., and Tappin, G. (1966). Proc. Brit. Ceram. Soc., (6), 163.

Coble, R. L., (1963), J. Appl. Phys., 34, 1679.

Copley, S. M. and Pask, J. A. (1965a). J. Am. Ceram. Soc., 48, 636.

Copley, S. M. and Pask, J. A. (1965b). J. Am. Ceram. Soc., 48, 139.

Cottrell, A. H. (1958). Trans. AIME, 212, 192.

Cropper, D. R. (1966). Report Number UCRL-17105, Lawrence Radiation Laboratory, University of California, Berkeley.

Cummerow, R. L. (1963). J. Appl. Phys., 34, 1724-1729.

Dahlberg, P., Carnahan, R. D., and Brittain, J. O. (1962). J. Appl. Phys.
33, 3493.

Daniels, A. U., Lowrie, R. C., Gibby, R. L., and Cutler, I. B. (1962).
J. Am. Ceram. Soc., 45, 282.

Davidge, R. W. (1967). J. Materials Science, 2, 339.

Davidge, R. W. and Tappin, G. (1967). Trans. Brit. Ceram. Soc., 66, 405.

Davies, M. O. (1963). J. Chem. Phys., 38, 2047.

Day, R. B. and Stokes, R. J. (1964). J. Am. Ceram. Soc., 47, 493.

Day, R. B. and Stokes, R. J. (1966a). J. Am. Ceram. Soc., 49, 72.

Day, R. B. and Stokes, R. J. (1966b). J. Am. Ceram. Soc. 49, 345.

Day, R. B. and Stokes, R. J. (1966c). Materials Sci. Res., 3, 355.

Day, R. B. and Stokes, R. J. (1967). J. Am. Ceram. Soc., 50, 445.

Desport, J. A. and Smith, J.A. G. (1964). J. Nucl. Materials, 14, 135.

Dew-Hughes, D. and Narlikar, A. V. (1967). J. Inst. Met., 95, 90.

de Wys, E. C. and Leipold, M. H. (1967). Materials Research Bulletin, 2,

Elkington, W. E., Thomas, G., and Washburn, J. (1963). J. Am. Ceram. Soc.,

46, 307.

Evans, P. R. V., (1963). Tech. Doc. Report Number ASD-TR-61-628, Part II,

(N. A. Weil, ed.), Illinois Institute of Technology, Chicago,

pp. 164-202.

Field, J. E. and Heyes, A. D. (1965). In "Kurzzeitphotographie," Proc.

Seventh International Conf. on High-Speed Photography (O. Helwich, ed.),

Zurich, pp. 391-398.

Gager, W. B., Klein, M. J., and Jones, W. H. (1964). Appl. Phys. Letters,

5, 131.

Gardner, R. E. and Robinson, G. W. (1962). J. Am. Ceram. Soc., 45, 46.

Ghosh, T. K. and Clarke, F. J. P. (1961). Brit. J. Appl. Phys., 12, 44.

Gifkins, R. C. (1968). J. Am. Ceram. Soc., 51, 69.

Gilman, J. J. (1959). Acta Met., 7, 608.

Gilman, J. J. (1961). Prog. in Ceramic Sci., 1, 146.

Gorum, A. E., Parker, E. R., and Pask, J. A. (1958). J. Am. Ceram. Soc.,

41, 161.

- Gorum, A. E., Luhman, W. J., and Pask, J. A. (1960). J. Am. Ceram. Soc. 43, 241.
- Groves, G. W. and Fine, M. E. (1964). J. Appl. Phys., 35, 3587.
- Groves, G. W. and Kelly, A. (1961). Phil. Mag., 6, 1527.
- Groves, G. W. and Kelly, A. (1962a). J. Appl. Phys., 33, 456.
- Groves, G. W. and Kelly, A. (1962b). Phil. Mag., 7, 892.
- Groves, G. W. and Kelly, A. (1963a). J. Appl. Phys., 34, 3104.
- Groves, G. W. and Kelly, A. (1963b). Proc. Roy. Soc., 275A, 233.
- Groves, G. W. and Kelly, A. (1963c). Phil. Mag., 8, 877.
- Groves, G. W. and Kelly, A. (1963d). Phil. Mag., 8, 1437.
- Groves, G. W. and Shockley, D. A. (1966). Tech. Report Number 2, Contract Number 760 (29), Carnegie Institute of Technology, Pittsburgh.
- Habraken, L. and Blavier, P. (1965). Silicates Industriels, 30, 117.
- Harding, B. C. (1967). Phil. Mag., 16, 1039.
- Harrison, W. B. (1964). J. Am. Ceram. Soc., 47, 574.
- Hart, P. E., Atkin, R. B. and Pask, J. A., to be published.
- Henderson, B. (1964). Phil. Mag., 9, 153.

Hensler, J. H. and Cullen, G. V. (1967). J. Am. Ceram. Soc., 50, 584.

Herring, C. (1950). J. Appl. Phys., 21, 437.

Hornstra, J. (1965). In "Science of Ceramics" (G. H. Stewart, ed.), Brit. Ceram. Soc., 2, 191.

Hulse, C. O. (1961). J. Am. Ceram. Soc., 44, 572.

Hulse, C. O. (1967). A.F.M.L. International Symposium, Dayton, Ohio.

Hulse, C. O., Copley, S. M., and Pask, J. A. (1963). J. Am. Ceram. Soc., 46, 317.

Hulse, C. O. and Pask, J. A. (1960). J. Am. Ceram. Soc., 43, 373.

Hulse, C. O. and Tice, W. K. (1965). Nature, 206, 79.

Hulse, C. O. and Tice, W. K. (1966). J. Am. Ceram. Soc., 49, 190.

Jacobson, L. A. and Fehrenbacher, L. L. (1966). AFML-TR-66-91, Wright-Patterson AFB, Ohio.

Janowski, K. R. and Rossi, R. C. (1967). J. Am. Ceram. Soc., 50, 599.

Johnston, T. L., Stokes, R. J., and Li, C. H. (1959). Phil. Mag., 4, 1316.

Johnston, T. L., Li, C. H., and Stokes, R. J. (1962a), In "Strengthening Mechanisms in Solids," pp. 341-374, Am. Soc. for Metals, Cleveland, Ohio.

Johnston, T. L., Stokes, R. J., and Li, C. H. (1962b). Phil. Mag., 7, 23.

Johnston, W. G. (1960). Phil. Mag., 5, 407.

Kamiya, Y. (1963). Japan J. Appl. Phys., 2, 386.

Kear, B. H., Taylor, A., and Pratt, P. L. (1959). Phil. Mag., 4, 665.

Keh, A. S. (1960). J. Appl. Phys., 31, 1538.

Keh, A. S., Li, J. C. M., and Chou, Y. T. (1959). Acta Met., 7, 694.

Kirkpatrick, H. B. and Amelinckx, S. (1962). Rev. Sci. Instrum., 33, 488.

Klein, M. J. (1965). Phil. Mag., 12, 735.

Klein, M. J. and Edington, J. W. (1966). Phil. Mag., 14, 21.

Klein, M. J. and Gager, W. B. (1966). J. Appl. Phys., 37, 4112.

Klein, M. J. and Rudman, P. S. (1966). Phil. Mag., 14, 1199.

Kohra, K. and Watanabe, H. (1961). J. Phys. Soc. Japan, 16, 580.

Kreglo, J. R. and Smothers, W. J. (1967). J. Am. Ceram. Soc., 50, 457.

Ku, R. C. and Johnston, T. L. (1964). Phil. Mag., 9, 231.

Kroupa, F. and Price, P. B. (1961). Phil. Mag., 6, 243.

Lad, R. A., Stearns, C. A., and Del Duca, M. G. (1958). Acta Met., 6,

- Lang, A. R. and Miles, G. D. (1965). J. Appl. Phys., 36, 1803.
- Lang, A. R. and Miuscov, V. F. (1964). Phil. Mag., 10, 263.
- Langdon, T. G. (1967). Rev. Sci. Instrum., 38, 125.
- Langdon, T. G. and Bell (1968), to be published.
- Langdon, T. G. and Pask, J. A. (1968a). In "Ceramic Microstructures - Their Analysis, Significance and Production," Proc. Third International Materials Symposium, University of California, Berkeley, (John Wiley and Sons, New York).
- Langdon, T. G. and Pask, J. A. (1968b), to be published.
- Lange, F. F. and Buessem, W. R. (1967). J. Appl. Phys., 38, 2013.
- Leipold, M. H. (1966). J. Am. Ceram. Soc., 49, 498.
- Leipold, M. H. (1967). J. Am. Ceram. Soc., 50, 628.
- Lewis, M. H. (1966a). Phil. Mag., 13, 777.
- Lewis, M. H. (1966b). Phil. Mag., 13, 1123.
- Lewis, M. H. (1966c). Proc. Conf. on the Physical Basis of Yield and Fracture, Oxford, Inst. of Physics and Physical Soc. (London), pp. 247-252.

- Lindner, R. and Parfitt, G. D. (1957). J. Chem. Phys., 26, 182.
- Liu, T. S., Stokes, R. J., and Li, C. H. (1964). J. Am. Ceram. Soc., 47,
276.
- Lowrie, R. (1963). Phil. Mag., 8, 1965.
- Matkin, D. I. and Bowen, D. H. (1965). Phil. Mag., 12, 1209.
- May, J. E. and Kronberg, M. L. (1960). J. Am. Ceram. Soc., 43, 525.
- McPherson, R. and Sinha, H. N. (1965). Phil. Mag., 11, 451.
- Miles, G. D. (1964a). Prog. Appl. Materials Research, 5, 127.
- Miles, G. D. (1964b). Acta Met., 12, 1241.
- Miles, G. D. (1965). J. Appl. Phys., 36, 1471.
- Miles, G. D. and Clarke, F. J. P. (1961). Phil. Mag., 6, 1449.
- Miles, G. D., Clarke, F. J. P., Henderson, B., and King, R. D. (1966).
Proc. Brit. Ceram. Soc., (6), 325.
- Miles, G. D., Sambell, R. A. J., Rutherford, J., and Stephenson, G. W.
(1967). Trans. Brit. Ceram. Soc., 66, 319.
- Miuscov, V. F. and Lang, A. R. (1963). Kristallografiya, 8, 652 (For
English translation, see Soviet Physics - Crystallography, 8, 520-523,
1964.)

- Mountvala, A. J. and Murray, G. T. (1966). Phil. Mag., 13, 441.
- Murray, G. T. (1960). J. Am. Ceram. Soc., 43, 330.
- Murray, G. T., Silgailis, J., and Mountvala, A. J. (1964). J. Am. Ceram. Soc., 47, 531.
- Nabarro, F. R. N. (1948). Report on Conf. on the Strength of Solids, pp. 75-90, Physical Soc., London.
- Narlikar, A. V. and Dew-Hughes, D. (1962-63). J. Inst. Met., 91, 377.
- Neiman, A. S. and Rothwell, W. S. (1963). Appl. Phys. Letters, 3, 160.
- Nicholson, G. C. (1966). J. Am. Ceram. Soc., 49, 47.
- Nielsen, T. H. and Leipold, M. H. (1963). J. Am. Ceram. Soc., 46, 381.
- Ogawa, K. (1966a). Phil. Mag., 13, 1097.
- Ogawa, K. (1966b). Phil. Mag., 14, 619.
- Oishi, Y. and Kingery, W. D. (1960). J. Chem. Phys., 33, 905.
- Orowan, E. (1959). In "Fracture" (B. L. Averbach et al., eds.), pp. 147-160, John Wiley and Sons, New York, and Technology Press of M.I.T.
- Parker, E. R. (1959). In "Fracture" (B. L. Averbach et al., eds), pp. 181-192, John Wiley and Sons, New York, and Technology Press of M.I.T.

Parker, E. R. (1961). In "Mechanical Properties of Engineering Ceramics"

(W. W. Kriegel and H. Palmour III, eds.), pp. 65-83, Interscience,

New York.

Parker, E. R., Pask, J. A., Washburn, J., Gorum, A. E., and Luhman, W.

(1958). J. Metals, 10, 351.

Pask, J. A. and Copley, S. M. (1963). In "High Temperature Structures

and Materials," pp. 57-94, Pergamon Press, Oxford.

Passmore, E. M., Duff, R. H., and Vasilos, T. (1966). J. Am. Ceram. Soc.,

49, 594.

Phillips, W. L. (1960). Trans. AIME, 218, 939.

Phillips, W. L. (1962). Trans. AIME, 224, 434.

Rhodes, W., Sellers, D., and Vasilos, T. (1967). Report Number AMRA CR

67-01(F), Avco Corporation, Lowell, Mass.

Rice, R. W. (1962), "Product of Transparent MgO at Moderate Temperatures

and Pressures," presented at the Sixtyfourth Annual Meeting, Am. Ceram.

Soc., New York, May 1, 1962.

Rice, R. (1963). Space/Aeronautics, pp. 109-112, May 1963.

Rice, R. W. (1966a). Materials Sci. Res., 3, 387.

Rice, R. W. (1966b). In "Ceramic Microstructures - Their Analysis, Significance and Production," Proc. Third International Materials Symposium, University of California, Berkeley, (John Wiley and Sons, New York.

Rice, R. W. and Hunt, J. G. (1966). Interim Report Number II, Contract NAS 7-276, The Boeing Company, Seattle, Washington.

Rice, R. W. and Hunt, J. G. (1967). Interim Report Number III, Contract NAS 7-276, The Boeing Company, Seattle, Washington.

Robins, J. L., Gerlach, R. L., and Rhodin, T. N. (1966). Tech. Report Number 11, Project Number NR 036-025, O.N.R., Washington, D.C.

Rothwell, W. S. (1966). J. Appl. Phys., 37, 2833.

Rothwell, W. S. and Neiman, A. S. (1965). J. Appl. Phys., 36, 2309.

Sambell, R. A. J. and Bradley, R. (1964). Phil. Mag., 9, 161.

Satkiewicz, F. G. (1961). In "Mechanical Properties of Engineering Ceramics" (W. W. Kriegel and H. Palmour III, eds.), pp. 49-61, Interscience, New York.

Sinha, H. N. (1963). Phil. Mag., 8, 2115.

Sinha, H. N. (1964). J. Aust. Inst. Met., 9, 184.

Sinha, H. N. (1965). Tewksbury Symposium on Fracture (1963), pp. 100-118,

Eng. Faculty, University of Melbourne.

Skinner, B. J. (1957). Am. Mineral., 42, 39.

Smyth, H. T. and Leipold, M. H. (1967). Tech. Report Number 32-1042,

J. P. L., Pasadena.

Spriggs, R. M. (1965). Twelfth Sagamore Army Materials Research Conference,

Raquette Lake, N. Y., August 1965.

Spriggs, R. M. and Vasilos, T. (1963). J. Am. Ceram. Soc., 46, 224.

Spriggs, R. M. and Vasilos, T. (1964). J. Am. Ceram. Soc., 47, 47.

Spriggs, R. M., Brissette, L. A., and Vasilos, T. (1962). J. Am. Ceram.

Soc., 45, 400.

Spriggs, R. M., Brissette, L. A., and Vasilos, T. (1963). J. Am. Ceram.

Soc., 46, 508.

Spriggs, R. M., Brissette, L. A., and Vasilos, T. (1964). J. Am. Ceram.

Soc., 47, 417.

Spriggs, R. M., Vasilos, T., and Brissette, L. A. (1966). Materials Sci.

Res., 3, 313.

Stablein, P. F. (1963). J. Appl. Phys., 34, 1867.

Steifbold, D. R., Boston, J. R., and Huggins, R. A. (1963). J. Am. Ceram.

Soc., 46, 507.

Stokes, R. J. (1962). Trans. AIME, 224, 1227.

Stokes, R. J. (1965a). J. Am. Ceram. Soc., 48, 60.

Stokes, R. J. (1965b). Twelfth Sagamore Army Materials Research Conference

on Strengthening Mechanisms in Metals and Ceramics, Sagamore, N.Y.

Stokes, R. J., (1966a). Proc. Conf. on Nuclear Applications of Non-

fissionable Ceramics, Washington, D. C., pp. 3-29, Am. Nucl. Soc.

Stokes, R. J. (1966b). J. Am. Ceram. Soc., 49, 39.

Stokes, R. J. and Li, C. H. (1963a). Materials Sci. Res., 1, 133.

Stokes, R. J. and Li, C. H. (1963b). J. Am. Ceram. Soc., 46, 423.

Stokes, R. J. and Li, C. H. (1964). Discs. Faraday Soc., 38, 233.

Stokes, R. J. and Olsen, K. H. (1963). Phil. Mag., 8, 957.

Stokes, R. J., Johnston, T. L., and Li, C. H. (1958). Phil. Mag., 3, 718.

Stokes, R. J., Johnston, T. L., and Li, C. H. (1959a). Phil. Mag., 4,

137.

Stokes, R. J., Johnston, T. L., and Li, C. H. (1959b). Trans. AIME, 215,

437.

Stokes, R. J., Johnston, T. L., and Li, C. H. (1959c). Phil. Mag., 4,

920.

Stokes, R. J., Johnston, T. L., and Li, C. H. (1960). Trans. AIME, 218, 655

Stokes, R. J., Johnston, T. L., and Li, C. H. (1961). Phil. Mag., 6, 9.

Stokes, R. J., Johnston, T. L., and Li, C. H. (1962). J. Appl. Phys.,

33, 62.

Stroh, A. N. (1954). Proc. Roy. Soc., 223A, 404.

Stroh, A. N. (1955). Proc. Roy. Soc., 232A, 548.

Stroh, A. N. (1957). Advances in Physics, 6, 418.

Tagai, H., Zisner, T., Mori, T., and Yasuda, E. (1967). J. Am. Ceram.

Soc., 50, 550.

Tattersall, H. G. and Clarke, F. J. P. (1962). Phil. Mag., 7, 1977.

Tattersall, H. G. and Tappin, G. (1966). J. Materials Science, 1, 296.

Thompson, D. S. and Roberts, J. P. (1960). J. Appl. Phys., 31, 433.

Uyeda, R. and Nonoyama, M. (1965). Japan J. Appl. Phys., 4, 498.

Vasilos, T. and Spriggs, R. M. (1963). J. Am. Ceram. Soc., 46, 493.

Vasilos, T. and Spriggs, R. M. (1965). Proc. Brit. Ceram. Soc., (3), 195.

Vasilos, T., Mitchell, J. B., and Spriggs, R. M. (1964a). J. Am. Ceram. Soc., 47, 203.

Vasilos, T., Mitchell, J. B., and Spriggs, R. M. (1964b). J. Am. Ceram. Soc., 47, 606.

Vasilos, T., Spriggs, R. M., Brissette, L. A., Burnett, P., and Passmore, E. M., (1965). Summary Report, Contract N0w 64-0217-d, Avco Corporation, Lowell, Mass.

Venables, J. D. (1961). Phys. Rev., 122, 1388.

Venables, J. D. (1963). J. Appl. Phys., 34, 293.

von Misés, R. (1928). Z. Angew. Math. Mech., 8, 161.

Wachtman, J. B. (1957). In "Creep and Recovery," pp. 344-360, Am. Soc. for Metals, Cleveland, Ohio.

Wachtman, J. B. (1967). Ceramic Bulletin, 46, 756.

Washburn, J. (1963). In "Electron Microscopy and the Strength of Crystals"

(G. Thomas and J. Washburn, eds.), pp. 301-332, Interscience, New York.

Washburn, J. (1967). In "An Atomistic Approach to the Nature and Proper-

ties of Materials," (Joseph A. Pask, ed.), pp. 103-135, John Wiley &

Sons, Inc., New York.

Washburn, J. and Cass, T. (1966). J. de Physique, 27, Colloque C3, 168.

Washburn, J. and Gorum, A. E. (1960). Mem. Sci. Rev. Met., 57, 67.

Washburn, J., Gorum, A. E. and Parker, E. R. (1959). Trans. AIME, 215,

230.

Washburn, J., Groves, G. W., Kelly, A., and Williamson, G. K. (1960a).

Phil. Mag., 5, 991.

Washburn, J., Kelly, A., and Williamson, G. K. (1960b). Phil. Mag., 5,

192.

Watanabe, H., Fukuhara, A., and Kohra, K. (1962). J. Phys. Soc. Japan,

17, Suppl. B-II, 195.

Weaver, C. W. (1967). Phil. Mag., 15, 177.

Weertman, J. (1955). J. Appl. Phys., 26, 1213.

Weertman, J. (1957). J. Appl. Phys., 28, 362.

Westwood, A. R. C. (1961a). Phil. Mag., 6, 195.

Westwood, A. R. C. (1961b). In "Mechanical Properties of Engineering
Ceramics" (W. W. Kriegel and H. Palmour III, eds.), pp. 89-91, Inter-
science, New York.

Williams, M. W. and Arakawa, E. T. (1967). J. Appl. Phys., 38, 5272.

Wolff, E. G. and Coskren, T. D. (1965). J. Am. Ceram. Soc., 48, 279.

Wuensch, B. J. and Vasilos, T. (1966). J. Am. Ceram. Soc., 49, 433.

Wygant, J. F. (1951). J. Am. Ceram. Soc., 34, 374.

Zener, C. (1948). In "Fracturing of Metals," pp. 3-31, Am. Soc. for
Metals, Cleveland, Ohio.

TABLES

Table I. Tests on MgO Bi-Crystals

Table II. Summary of Fabrication Process (Miles et al. 1967).

Table III. Creep of Magnesium Oxide.

Table I. Tests on MgO Bi-crystals

| Type of boundary | Type of test | Temperature | Results | Reference |
|---------------------------|-----------------|-------------------------|---|-----------------------------|
| 10°-20° tilt <5° twist | Compression | Room temp. | Cracks formed when two slip bands meet at boundary | Westwood (1961a, 1961b) |
| - | Thermal shock | - | Possibility of crack nucleation at boundary | Clarke et al. (1961) |
| ~10° misorientation | Thermal shock | - | Grain boundary is source of weakness | Miles and Clarke (1961) |
| Tilt + twist | Compression | 1355-1495°C | Grain boundary sliding strongly dependent on boundary misorientation | Adams and Murray (1962) |
| Large tilt + large twist | 3-point bending | Room temp. | Intergranular crack formed | Johnston et al. (1962) |
| Tilt + twist | 3-point bending | Room temp. | Cracks produced at boundary, probably by Zeher mechanism | Clarke et al. (1962a) |
| Simple tilt | 3-point bending | Room temp. | Slip bands nucleated in adjacent grain | Johnston et al. (1962b) |
| Tilt + twist | 3-point bending | Room temp. | Transgranular crack nucleated in adjacent grain | Johnston et al. (1962b) |
| ~10° misorientation | 3-point bending | Room temp. | Cracks nucleated along slip planes due to dislocation pile-up | Sinha (1963) |
| Misorientations | Tension | Room temp. | Strength critically dependent on surface condition | Stokes and Li (1963b) |
| Various misorientations | 4-point bending | Room temp. up to 1097°C | Stress measured to form crack at tip of blocked glide band | Ku and Johnston (1964) |
| Tilt + twist | Compression | 1300-1500°C | Grain boundary sliding recorded as function of temperature and shear stress | Murray et al. (1964) |
| Various misorientations | 3-point bending | Room temp. | Silicate phase at boundary is major source of weakness | McPherson and Sinha (1965) |
| Simple tilt (<5°) | Bending | Room temp. | Slip crossed boundary without nucleating crack | Sinha (1965) |
| Tilt + twist | Bending | Room temp. and 1000°C | Transgranular fracture | Sinha (1965) |
| Large misorientations | Bending | Room temp. and 1000°C | Intergranular fracture. Some plastic deformation at 1000°C | Sinha (1965) |
| High tilt + low twist | Compression | 1200-1500°C | Catastrophic drop in fracture strength at ~1400°C | Mountvala and Murray (1966) |
| Medium tilt + high twist | Compression | 1200-1500°C | Catastrophic drop in fracture strength at ~1200°C | Mountvala and Murray (1966) |
| Various misorientations | Tension | Room temp. | Brittle fracture at strengths of 70,000-370,000 psi | Lange and Buessem (1967) |

Table II. Summary of Fabrication Process

| Stage | Details |
|--------------------------------|--|
| Raw materials | Mallinckrodt A. R. basic magnesium carbonate and laboratory-produced lithium fluoride. |
| Calcination of basic carbonate | Rotary calciner. 750°C bed temperature. Throughput 300 g/h. Surface area of powder 150-200 m ² /g. |
| Blending | 1% of LiF in isopropanol added to magnesia in isopropanol by shaking slurries together or by colloid milling (mill-stones set at zero clearance, slurry passed 20 times). |
| Drying | Carried out immediately after milling. Stirred continuously. Dried to approximately 10% alcohol content. |
| Pre-sintering (optional) | The MgO/LiF powder is compacted into a pure alumina tube mould of suitable size and heat-treated in air at 770°C to give a sintered cylindrical body just fitting the die. |
| Hot-pressing | Powder or pre-sintered body loaded direct into EY9 graphite die. Pre-pressed between the hands. Vacuum pressure <10 ⁻⁴ torr before starting programme. Programme stopped if pressure rises above 10 ⁻³ torr during cycle. Heating-rate, 200°C/h. 1500 p.s.i. applied at 770°C. Temperature held steady for 15 min. at 1000°C. Load removed when die cooled to 800°C. |
| Post-sintering | Furnace chamber large compared to specimen, or flowing air. Heating-rate, 30°C/h to 1200°C. 3 h soaking period. Residual mistiness or coloration can be removed by a longer soaking-period. |

TABLE III. Creep of magnesium oxide

| Percentage of theoretical density | Grain size | Type of test | Temperature range | Activation energy (kcal/mole) | Stress exponent, n | Suggested rate-controlling mechanism | Reference |
|-----------------------------------|------------|-----------------|-------------------|-------------------------------|----------------------|--|---|
| SINGLE CRYSTAL: | | | | | | | |
| | | 3-point bending | 1450-1700°C | 81-162 | 4-7 (1700-1500°C) | Climb of edge dislocations | Cummerow (1963) |
| | | 3-point bending | ~1000°C | 33.3 ± 5.7 | 5.2 ± 1.2 | Cross-slip of screw dislocations | Rothwell and Neiman (1965) |
| | | 3-point bending | 1300-1630°C | 134.5 ± 16.8 | 3 | Climb of edge dislocations | Rothwell and Neiman (1965) |
| | | Compression | 1200°C | - | 4.7 | Dissolution of dislocation loops and dipoles | Cropper (1966) |
| POLYCRYSTAL: | | | | | | | |
| 88% | - | Torsion | 1100-1300°C | 46.8 | 2.5 | - | Wygant (1951) ¹ |
| 98% | - | Torsion | 1100-1300°C | 46.4 | 3.5 | - | Wygant (1951) ² |
| -* | 1-3 μm | 4-point bending | 1180-1260°C | 74 | 1 | Nabarro-Herring or grain boundary diffusion | Vasilos et al. (1964a) ³ |
| >99.5% | 2 μm | 4-point bending | 1107-1527°C | 96.0 | 1.5 | Vacancy formation | Passmore et al. (1966) ³ |
| >99.5% | 5.5-20 μm | 4-point bending | 1107-1527°C | 54.1 | 1 | Vacancy formation | Passmore et al. (1966) ³ |
| 97% | 50 μm | 4-point bending | ~1650-1800°C | 89 | - | - | Bakunov et al. (1966) ⁴ |
| (Low) | - | Compression | 1205-1425°C | 62 | 4.5 | Dislocation mechanism | Kreglo and Smothers (1967) ⁵ |
| >99.5% | 12-52 μm | Compression | 1200°C | - | 3.3 | Climb of edge dislocations | Langdon and Pask (to be published) ⁶ |

* Specimens stated to be of "essentially full density."

1. Slip-cast MgO; 99.3% purity
2. Hydrostatically-pressed MgO; 99.3% purity
3. Hot-pressed MgO; 99.8% purity
4. Sintered MgO; 99.7% purity
5. Specimens cut from periclase bricks (98.2% MgO, 0.9% CaO, 0.5% SiO₂)
6. Hot-pressed MgO with a LiF additive; >99.8% purity

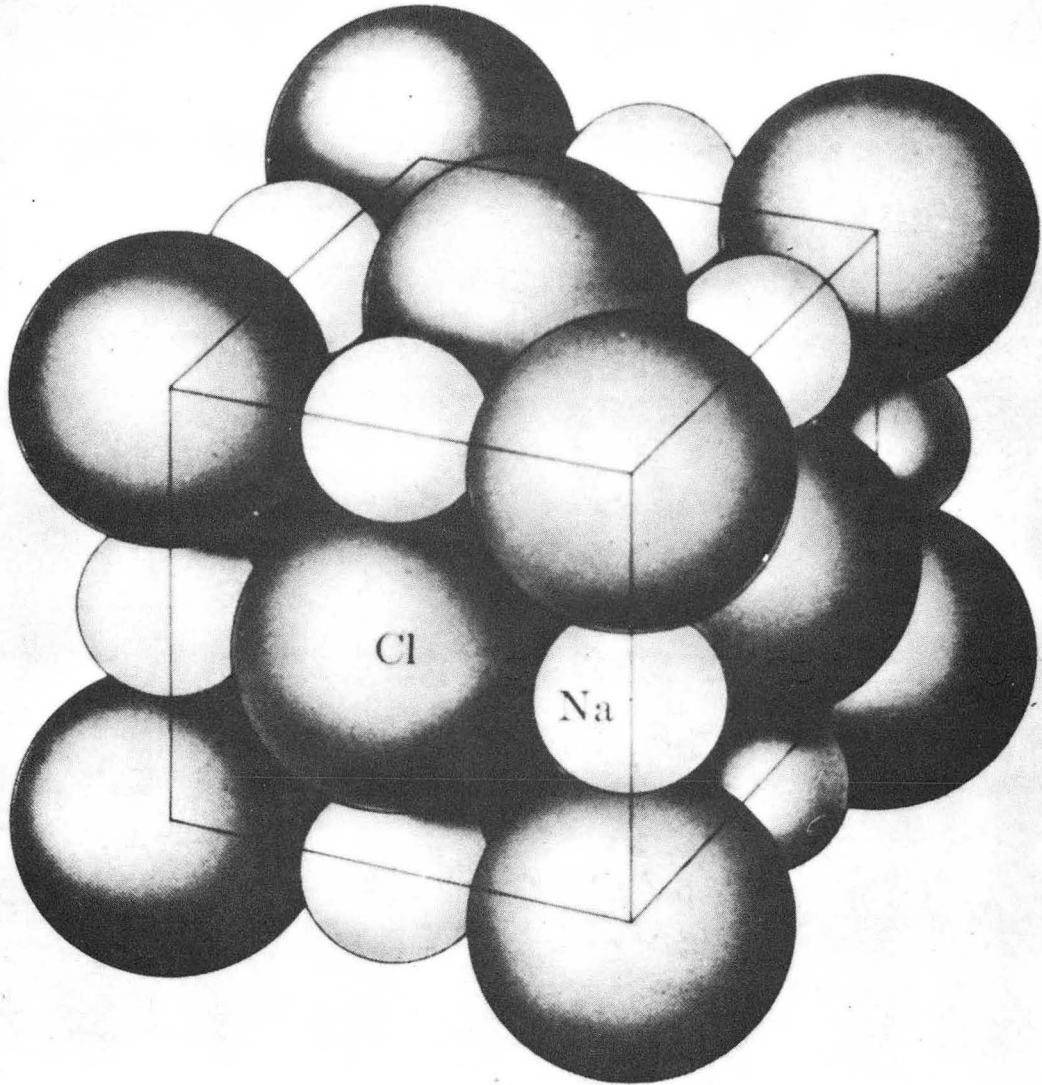
FIGURE CAPTIONS

- Fig. 1. Magnesium oxide structure--same as that for NaCl. Large spheres represent oxygen ions; small spheres represent magnesium ions.
- Fig. 2. Translation gliding in the same direction, but on two different planes in MgO crystals (Gilman, 1959).
- Fig. 3. (a) A diagram of the six slip planes of the $\{110\}$ $\langle 110 \rangle$ slip systems. (b) A diagram of the six Burgers vectors $\langle 110 \rangle$.
- Fig. 4. Etched rosette of dislocations at the surface of an MgO crystal.
- Fig. 5. Slip lines originating from rosettes in a lightly strained MgO crystal (Stokes et al. 1958).
- Fig. 6. (a) The distribution of dislocations around a kink band. (b) The position and orientation of the Stroh and secondary cracks around the kink band. (Stokes et al. 1958).
- Fig. 7. The location of slits with respect to bands of slip (Stokes et al. 1959a).
- Fig. 8. Suggested mechanism of microcrack growth (Clarke et al. 1962b).
- Fig. 9. Tensile stress-strain curves for chemically polished MgO crystals tested at room temperature. (Stokes et al., 1961).
- Fig. 10. The formation of a crack at the intersection of two expanding slip bands to give the distribution of slip in the composite photomicrograph which shows the location of the (110) $[001]$ slit fracture source (Stokes et al., 1961).
- Fig. 11. Stress-strain curves for MgO single crystals compressed with a $\langle 100 \rangle$ stress axis at various temperatures.
- Fig. 12. Stress-strain curves for MgO single crystals compressed with a $\langle 111 \rangle$ stress axis at various temperatures.

- Fig. 13. Specimen deformed 14.8% at 1400°C. (a) Illuminated by reflected light, and (b) between crossed polaroids, illuminated by transmitted light.
- Fig. 14. Diagrammatic representation of the various modes of deformation identified between 1000° and 2000°C on loading in tension. (Day and Stokes, 1964b).
- Fig. 15. Interaction of dislocations on conjugate slip systems. (a) Jog on screw dislocation, and (b) jog on edge dislocation. (Day and Stokes, 1964b).
- Fig. 16. Interaction of dislocations on 60° systems. (a) Dislocations E_1S_1 and E_2S_2 interact to produce a sessile dislocation E_3 along $\langle 11\bar{1} \rangle$, and (b) wall of sessile edge dislocations E_3 forming a kink boundary lying in the (110) plane parallel to tensile axis, σ . (Day and Stokes, 1964b).
- Fig. 17. Grown-in dislocation which does not lie on a slip plane. Dashed lines show approximately the projection of a {110} plane (Elkington et al., 1963).
- Fig. 18. Dislocation network in MgO crystal elongated 100% at 1850°C. (Stokes and Li, 1964).
- Fig. 19. {110} slip plane section of a MgO crystal deformed at 850°C. The gradual change of loop diameter in several of the narrow strings of loops is evidence for non-uniform spacing in the original dipole. (Washburn and Cass, 1966).
- Fig. 20. Boundary shear fracture stress as a function of temperature. (a) Low twist-high tilt boundary. (b) High twist-medium tilt boundary (Mountvala and Murray, 1966).

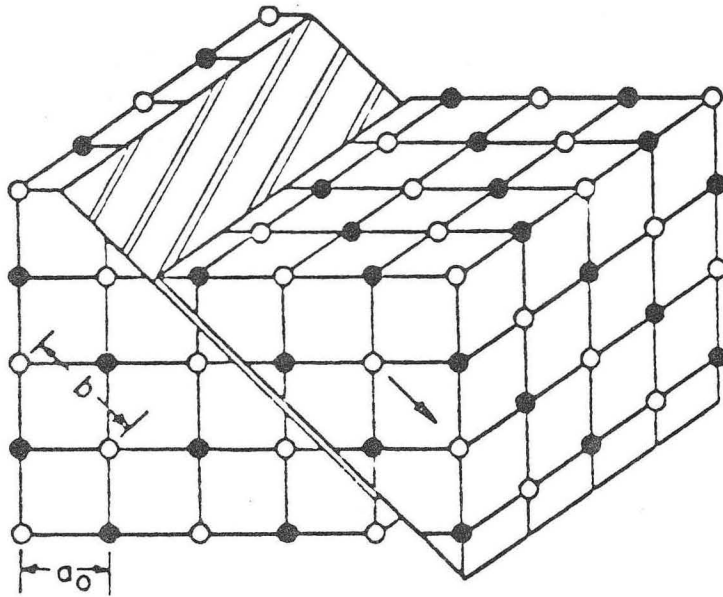
- Fig. 21. Cracks formed at grain boundary by piled-up groups of edge dislocations (Westwood, 1961a).
- Fig. 22. Fracture of bi-crystals under tension. (a) The intersection of a slip band with a grain boundary to generate a cleavage crack. (b) The three stages of fracture (i) cleavage crack nucleation, (ii) cleavage crack growth, and (iii) intergranular rupture. (Stokes and Li, 1963b).
- Fig. 23. Crack nucleation in a bi-crystal under compression. (Stokes and Li, 1963b).
- Fig. 24. Relative density vs. temperature curves obtained at a stress of 1700 psi and a heating rate of 5.5°C/min. (A = MgO powder, B = MgO powder plus 2 wt % LiF, and C = MgO powder presintered at 1400°C for 5 hrs plus 2 wt % LiF.)
- Fig. 25. Polycrystalline MgO disks: (left) hot-pressed with a LiF additive and subsequently heat treated, and (right) commercially sintered specimen. Microstructures of specimens shown below them.
- Fig. 26. Densification curves for the hot-pressing of various lithium compound additions to MgO. (Rhodes et al. 1967).
- Fig. 27. Stress-strain curves in tension for recrystallized polycrystalline MgO as a function of temperature (Day and Stokes, 1966b).
- Fig. 28. Surface folds due to slip in vicinity of triple points. (Day and Stokes, 1966b).

- Fig. 29. Yield stress vs temperature for the $\{110\}$ $\langle 1\bar{1}0 \rangle$ and $\{001\}$ $\langle 1\bar{1}0 \rangle$ slip systems (single crystals with $\langle 100 \rangle$ and $\langle 111 \rangle$ stress axes) and for type 1 and type 2 polycrystalline specimens.
- Fig. 30. Grain boundary shearing in type 1 polycrystalline MgO strained 3% at 1400°C .
- Fig. 31. Transmission electron micrograph of a section of polycrystalline MgO, showing introduction of dislocations into dislocation-free grain by cutting of the foil at point A.
- Fig. 32. (Top) Etch-pit pattern showing discrete slip bands formed by creep of MgO at 1000°C . (Bottom) Etch-pits in MgO showing polygonized structure after creep at 1600°C . Edges of both photos are along the $\langle 100 \rangle$ direction. (Rothwell and Neiman, 1965).

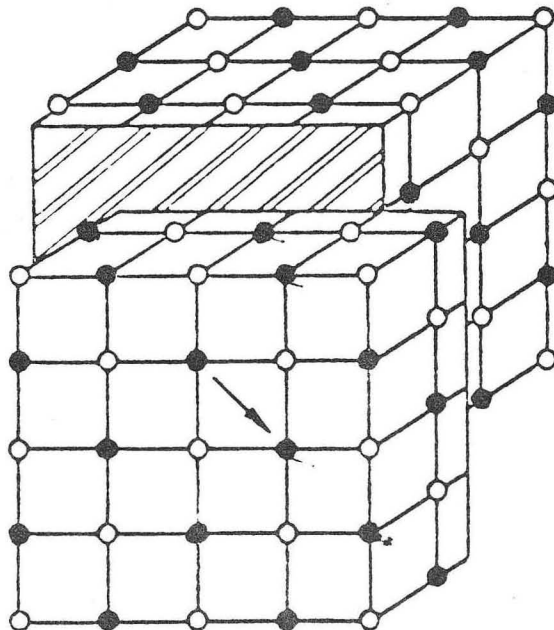


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Fig. 1



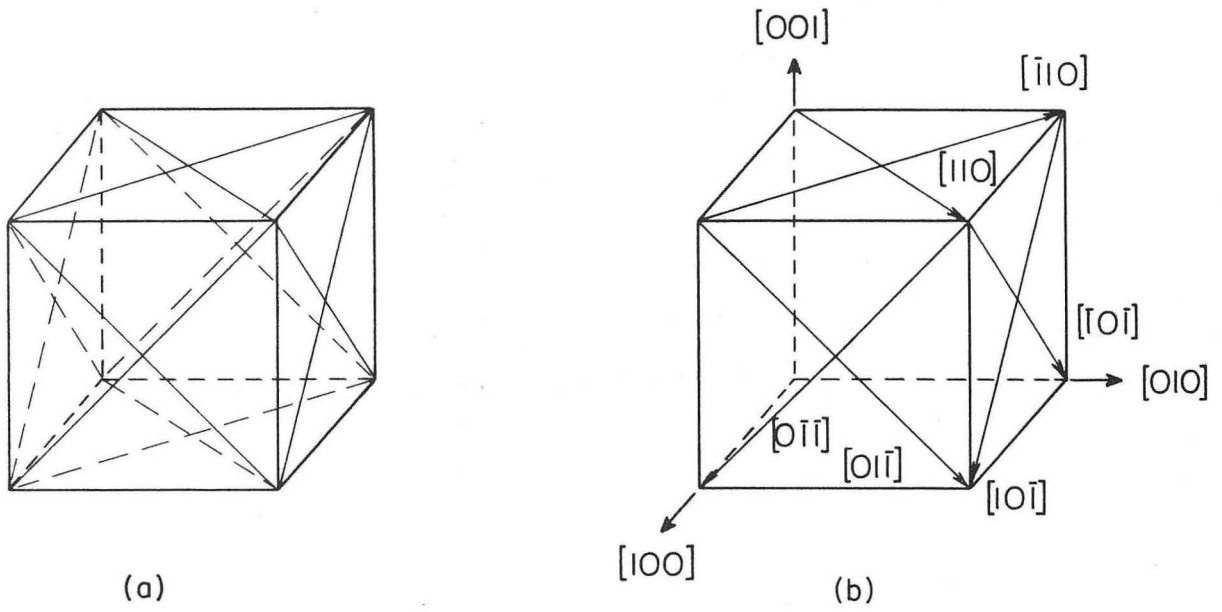
$\{110\} \langle 110 \rangle$ GLIDE



$\{100\} \langle 110 \rangle$ GLIDE

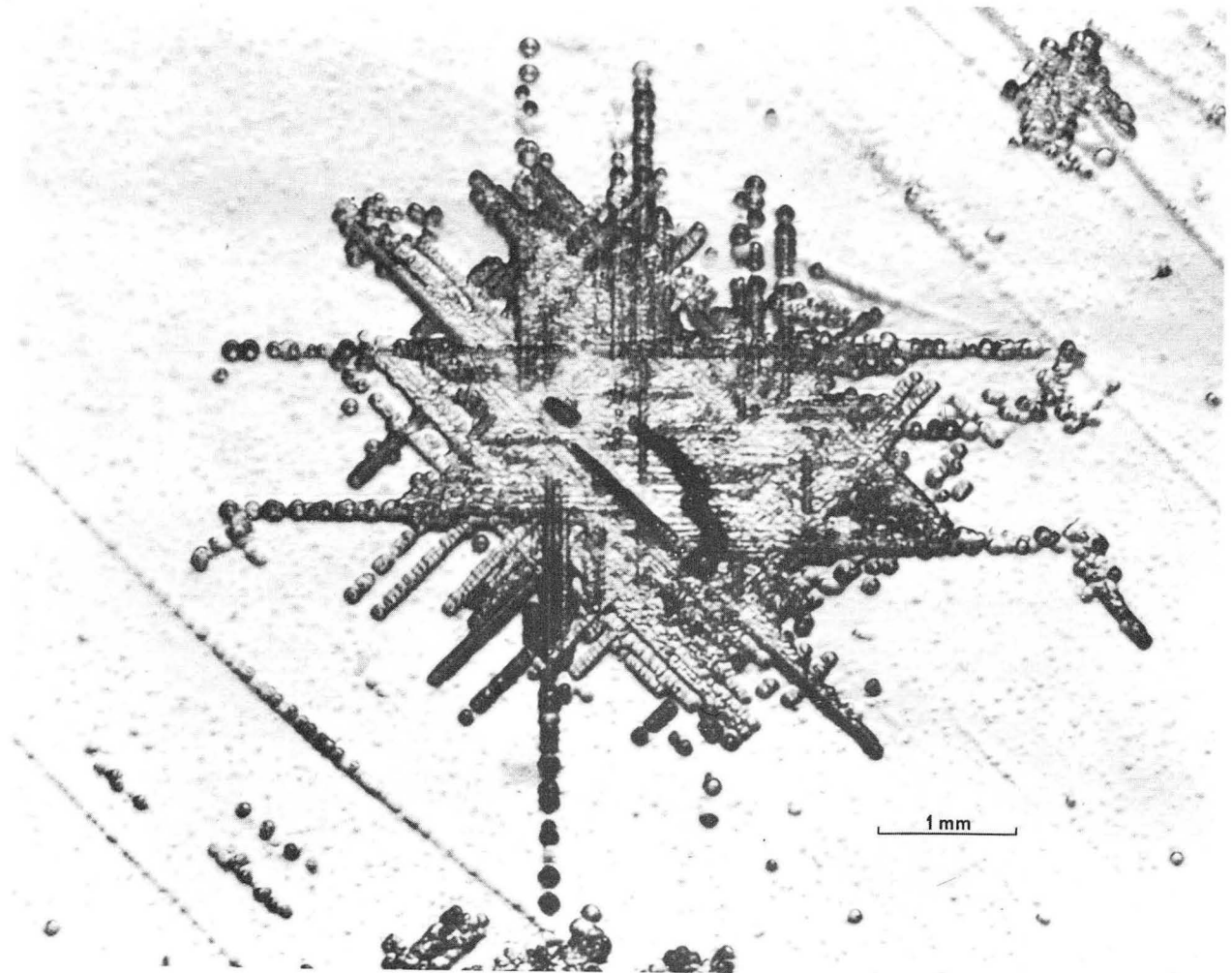
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Fig. 2



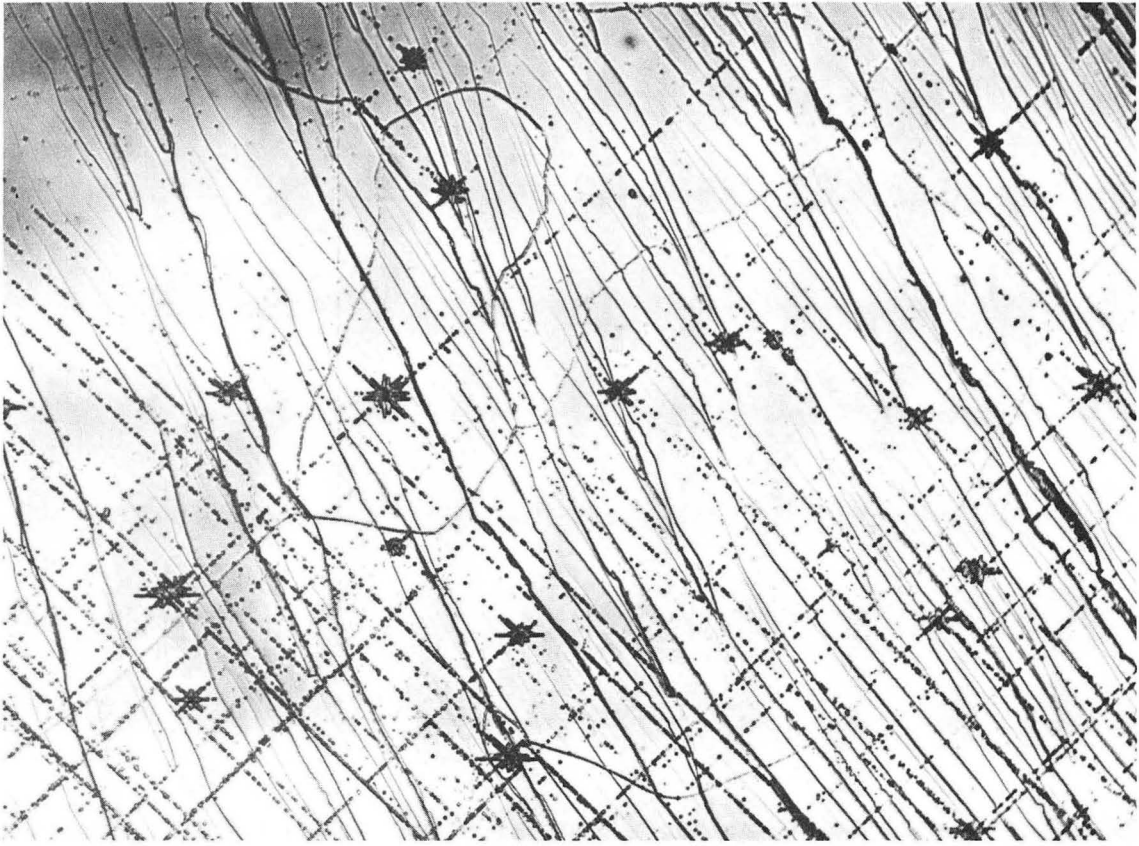
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Fig. 3



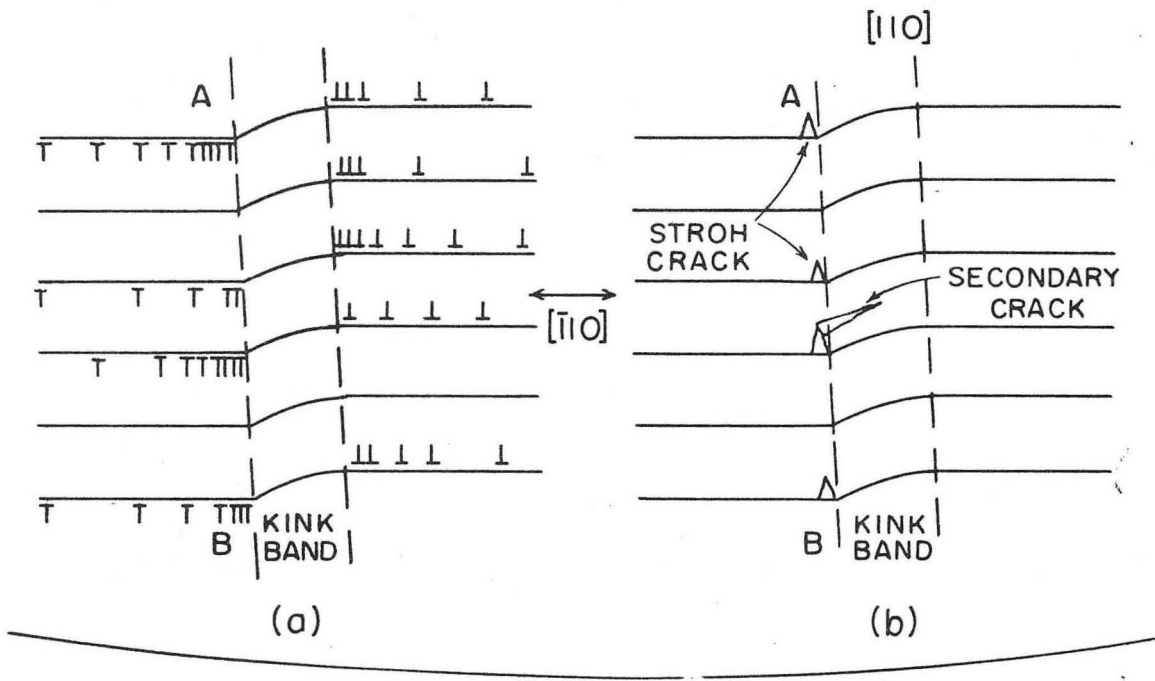
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Fig. 4



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Fig. 5



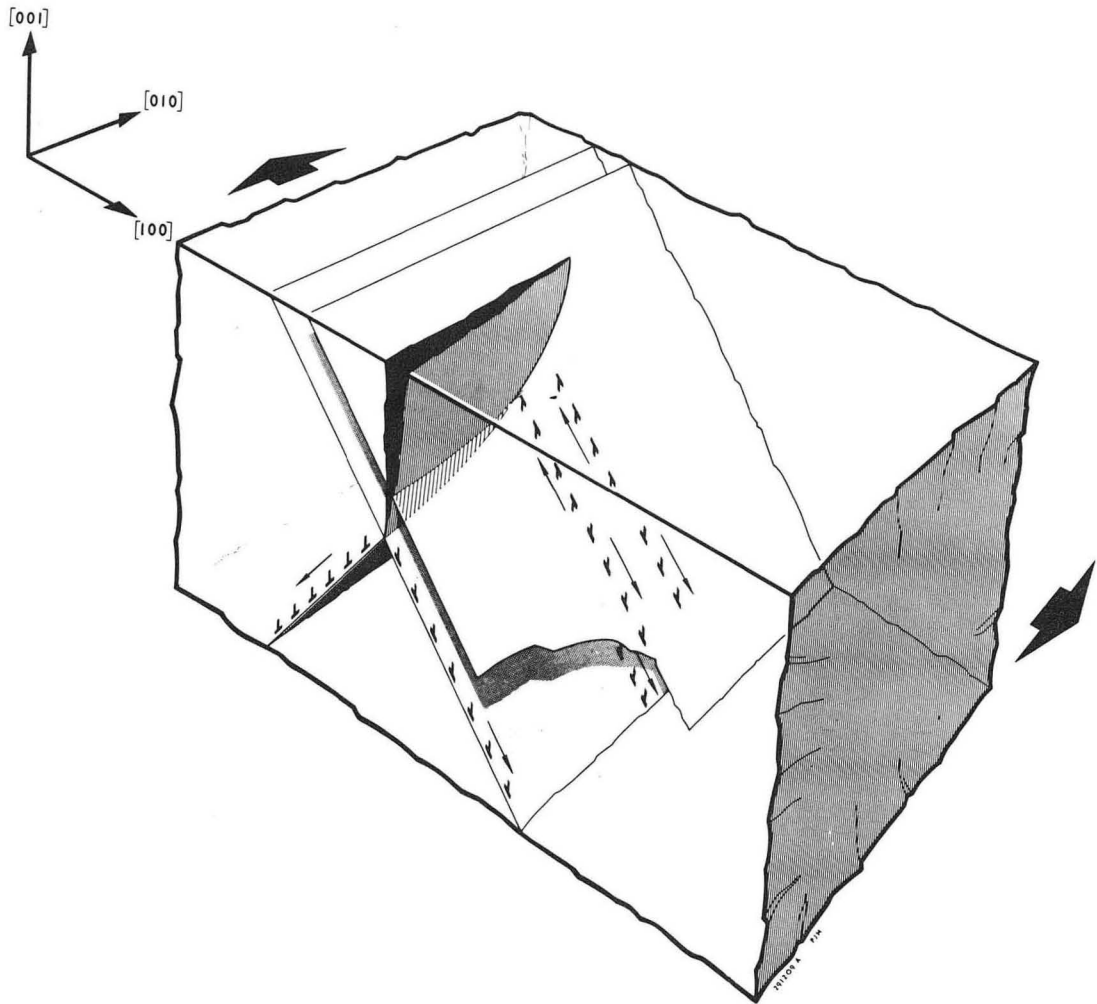
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Fig. 6



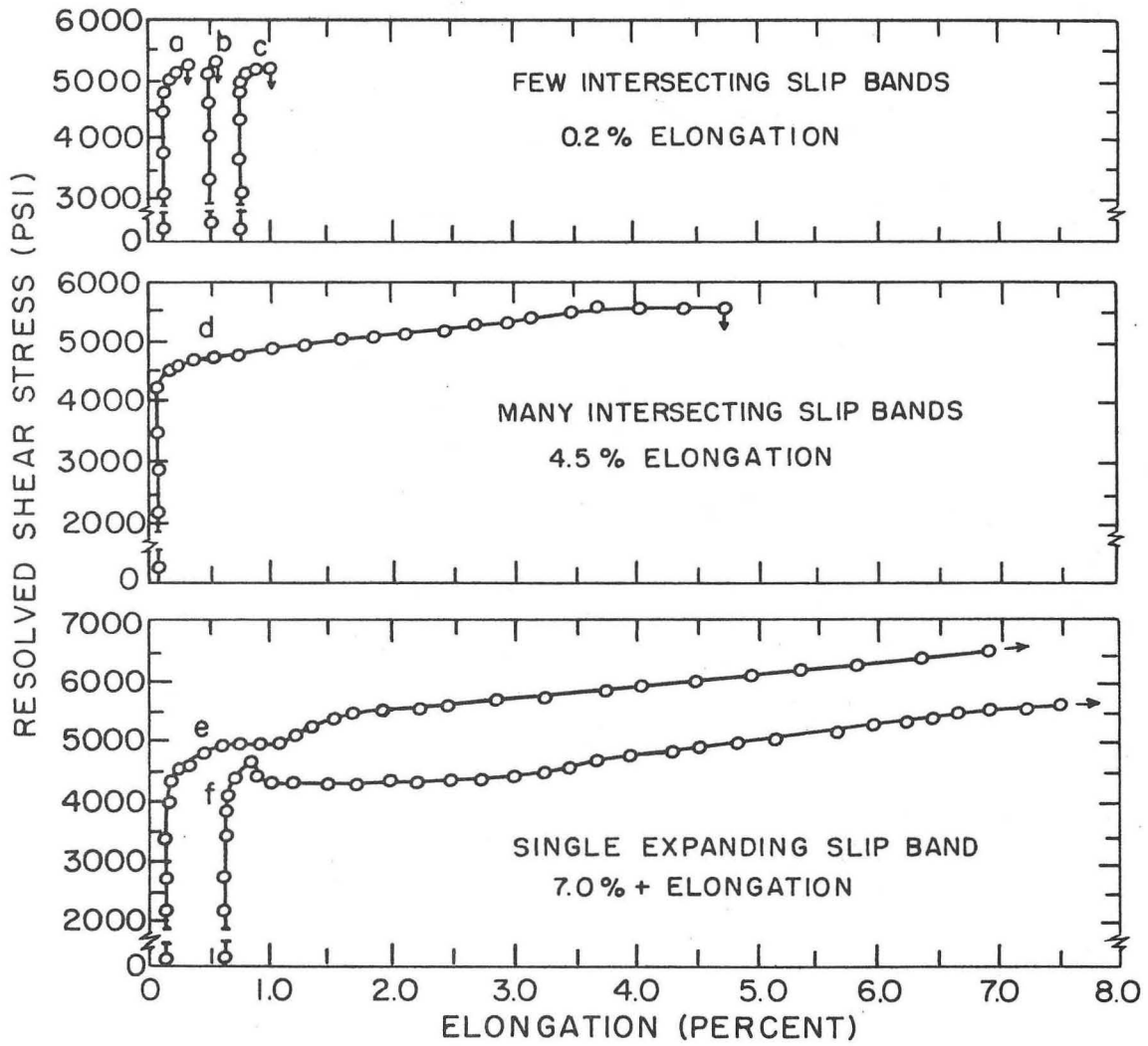
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Fig. 7



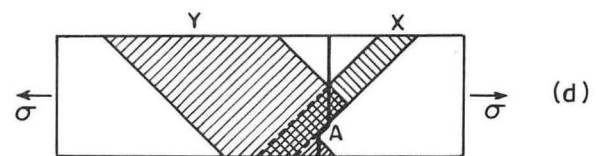
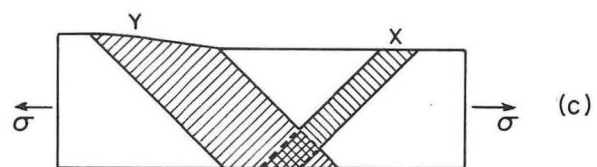
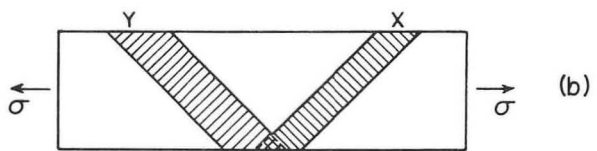
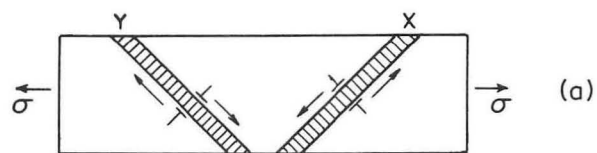
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Fig. 8

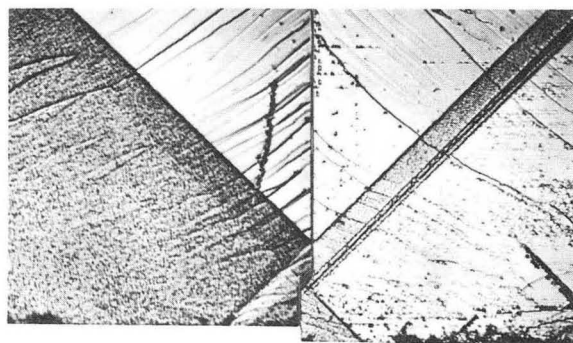


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Fig. 9

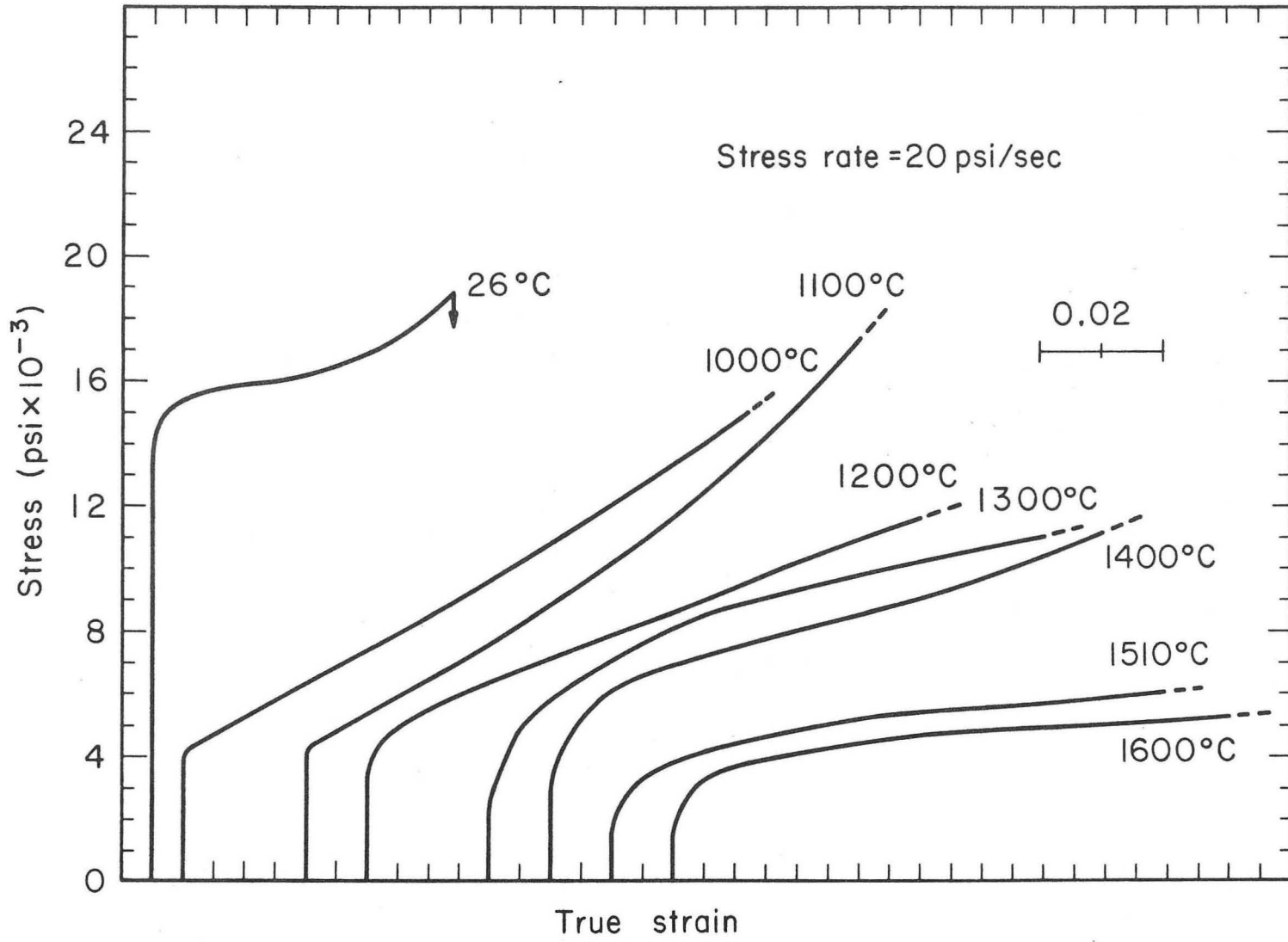


FRACTURE
PATH



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Fig. 10



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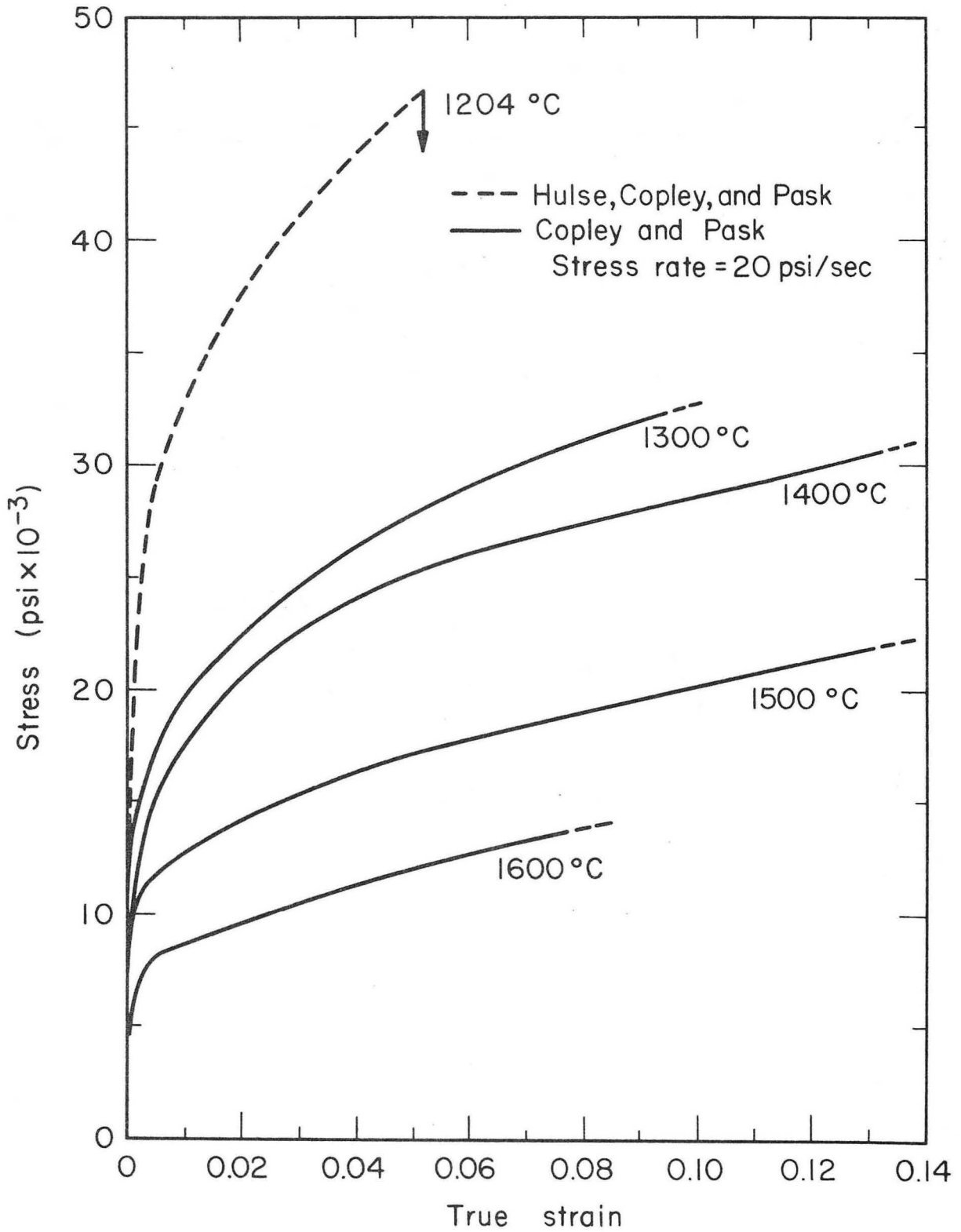
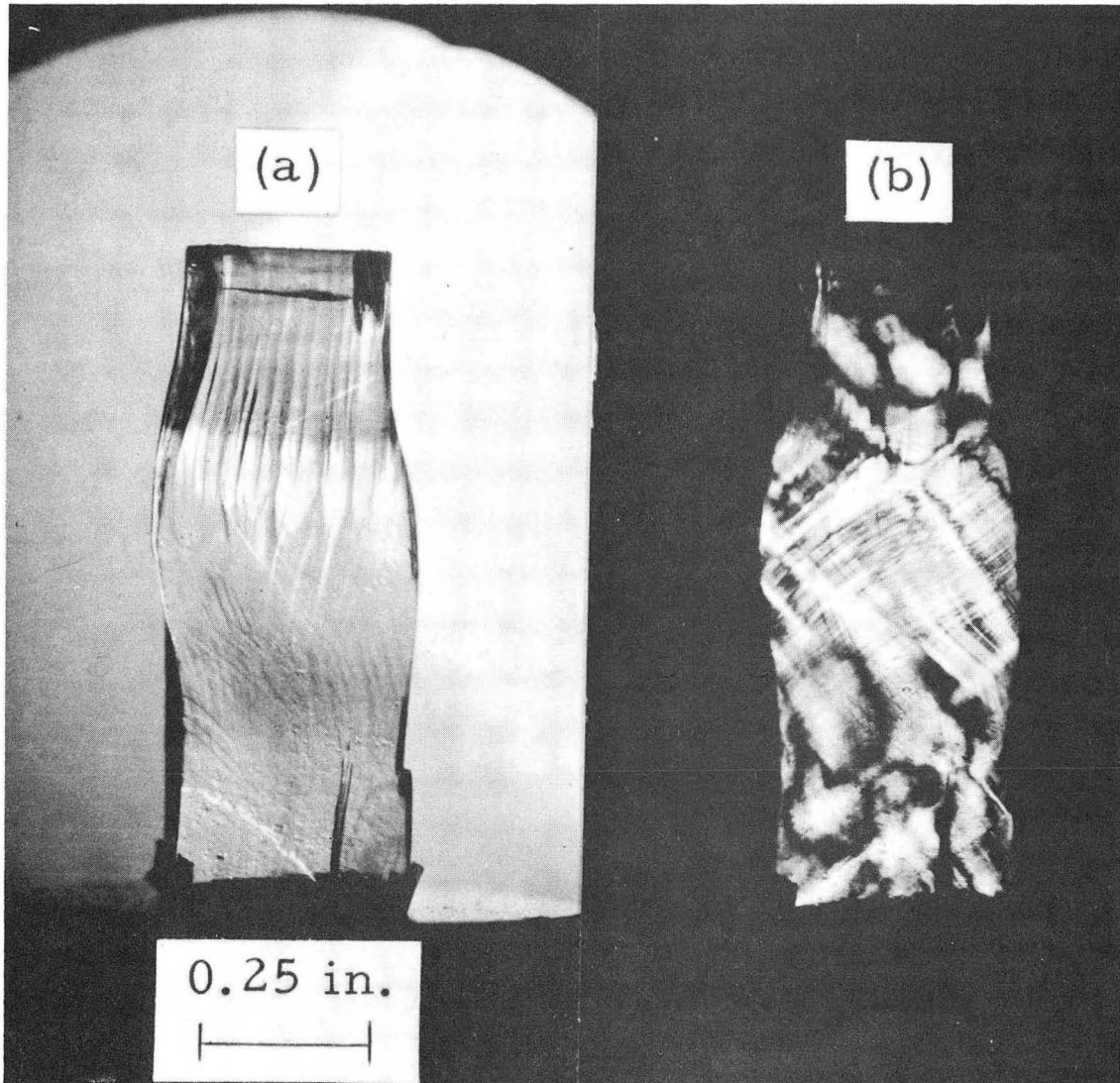
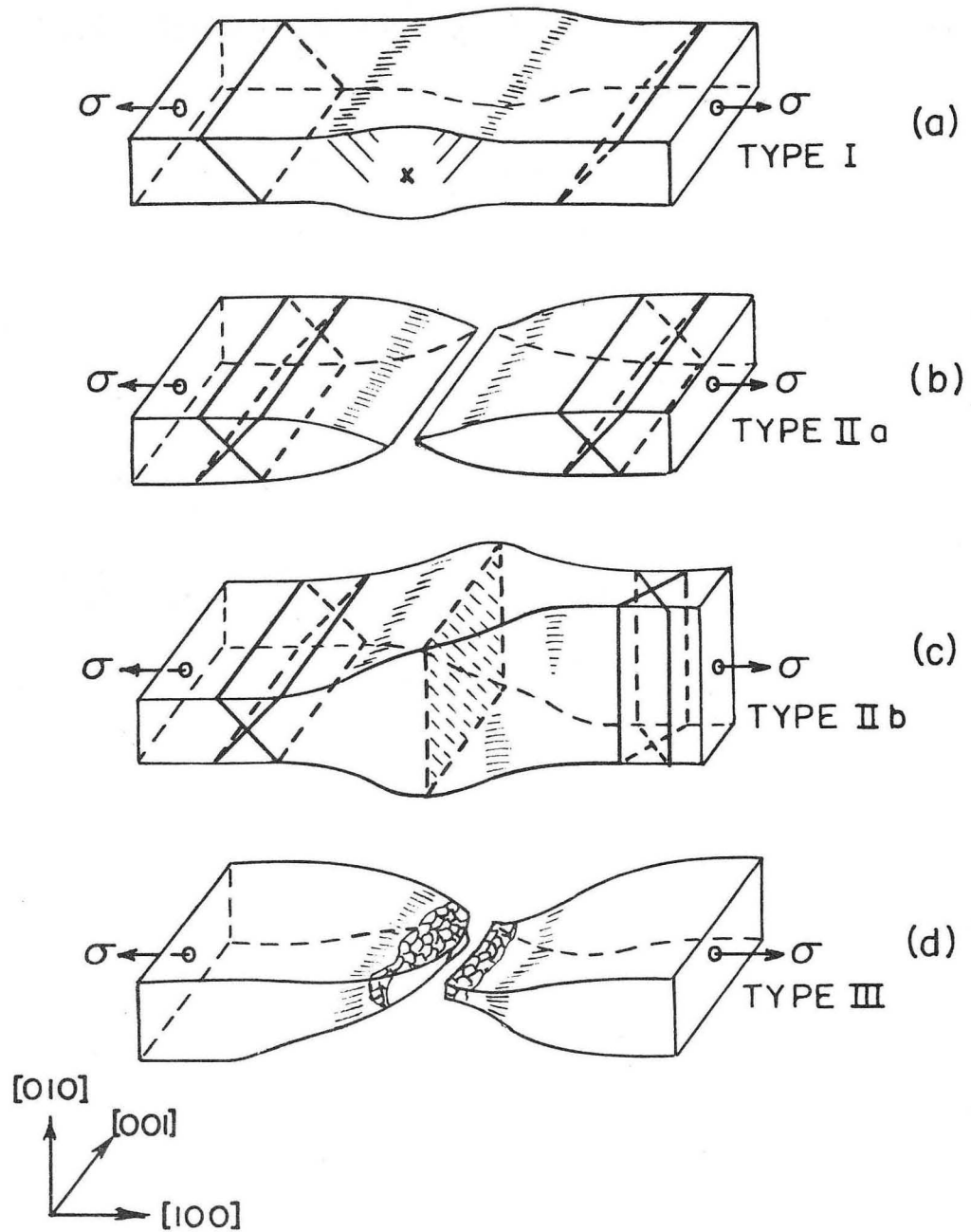


Fig. 12



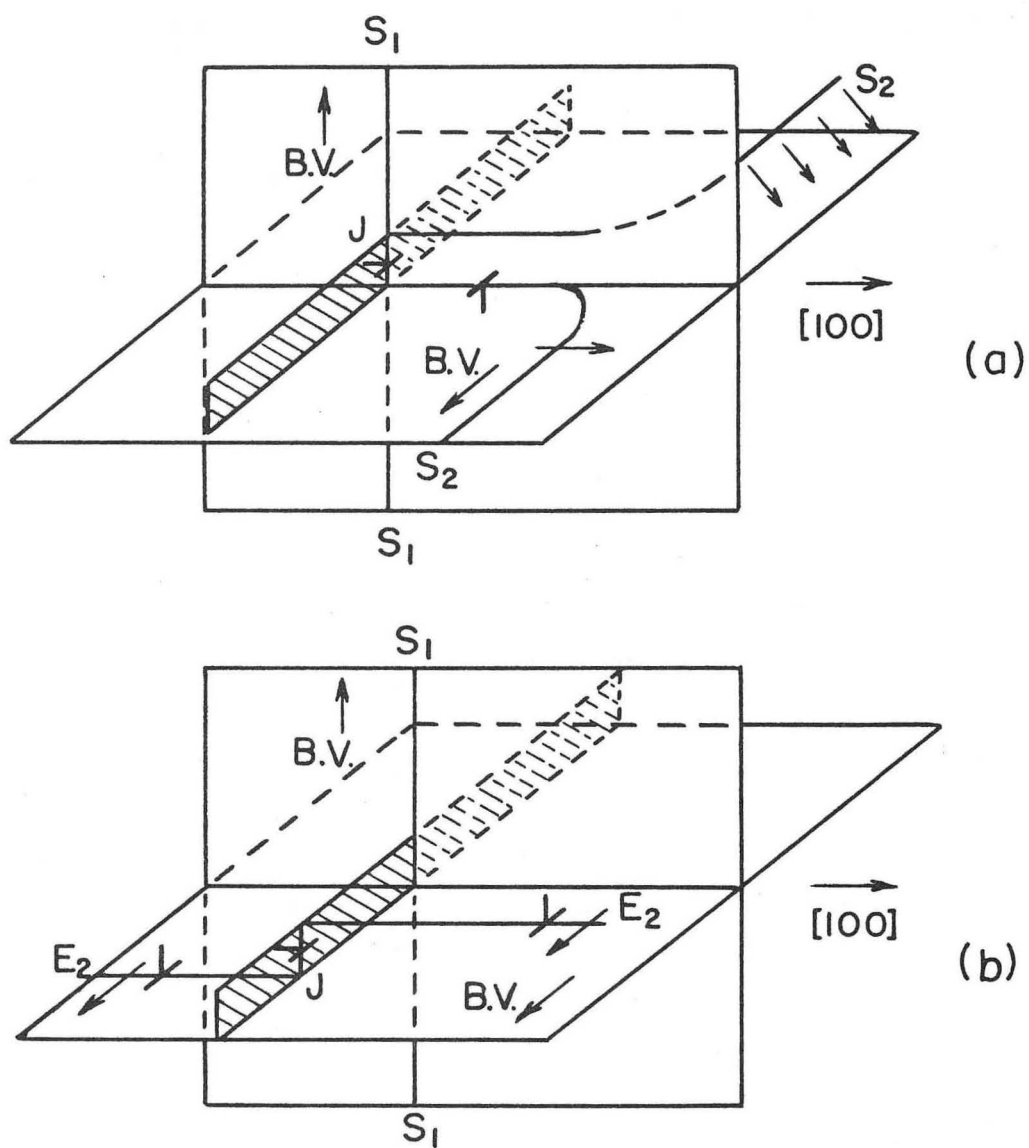
IM-987

Fig. 13



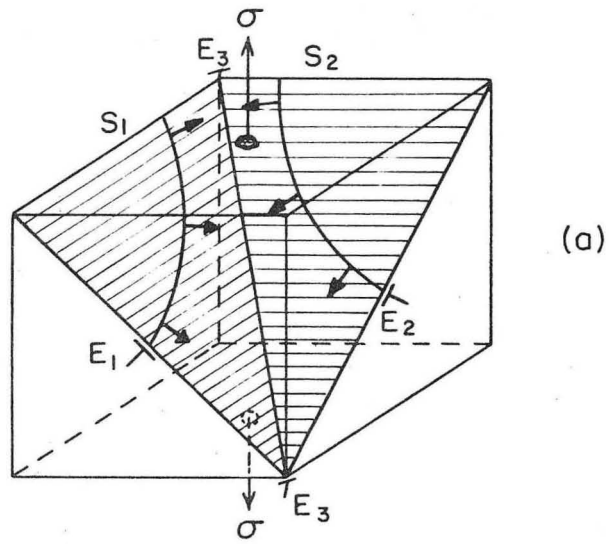
XBL 686-1112

Fig. 14

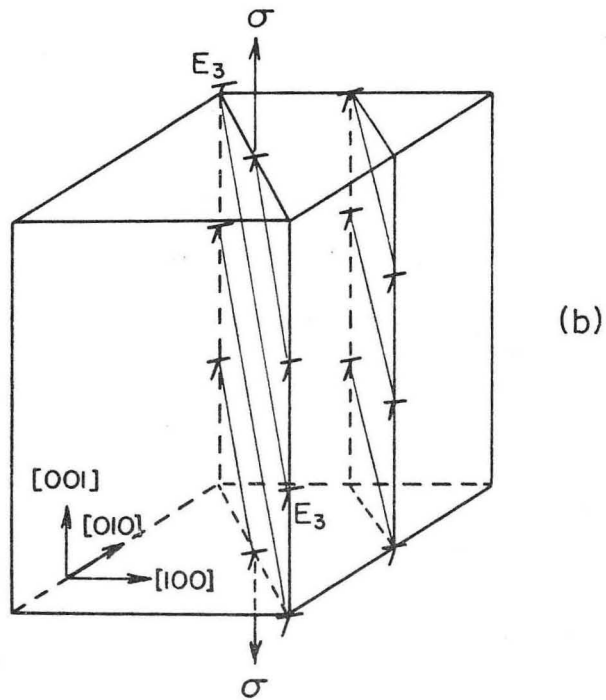


XBL 686-1113

Fig. 15



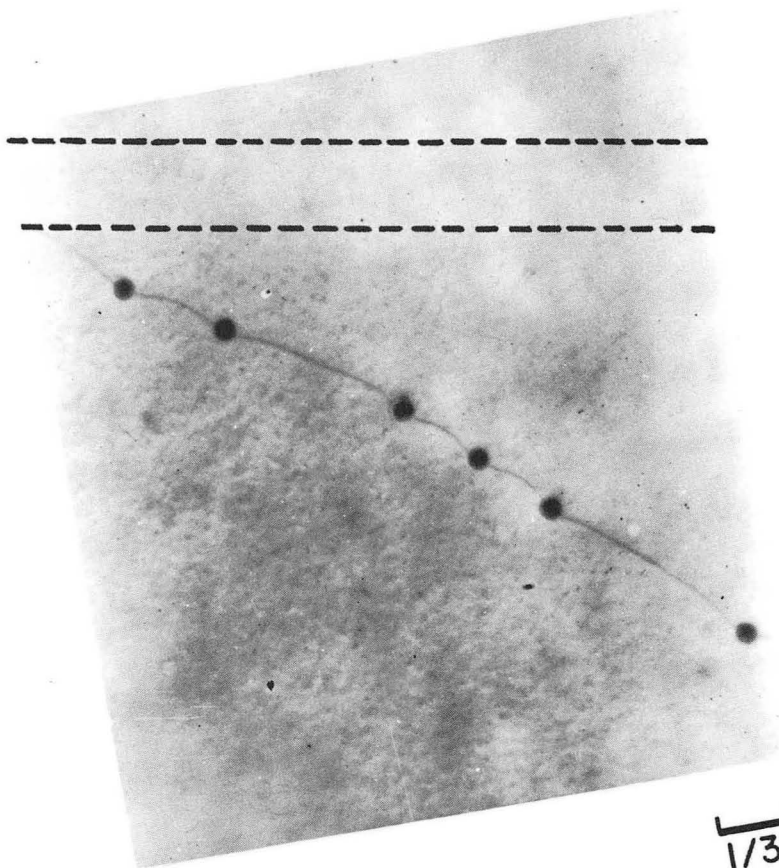
(a)



(b)

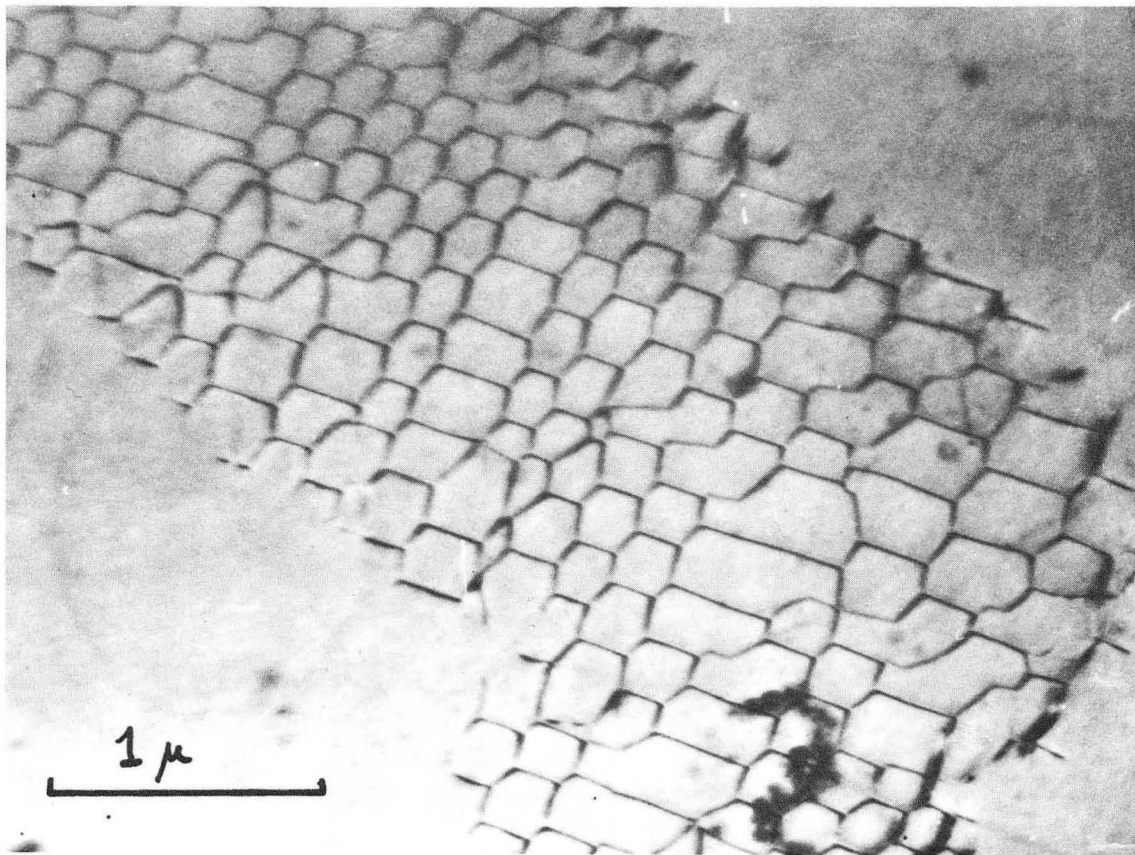
XBL 686-1114

Fig. 16



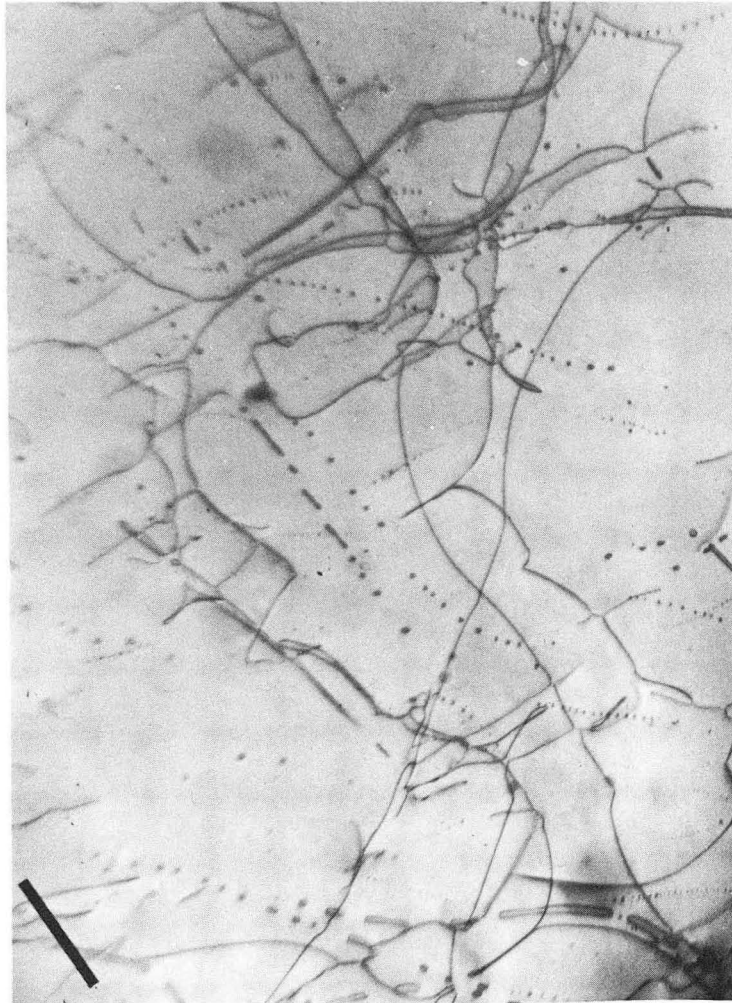
ZN-3161

Fig. 17



XBB 686-3950

Fig. 18



ZN-4994

Fig. 19

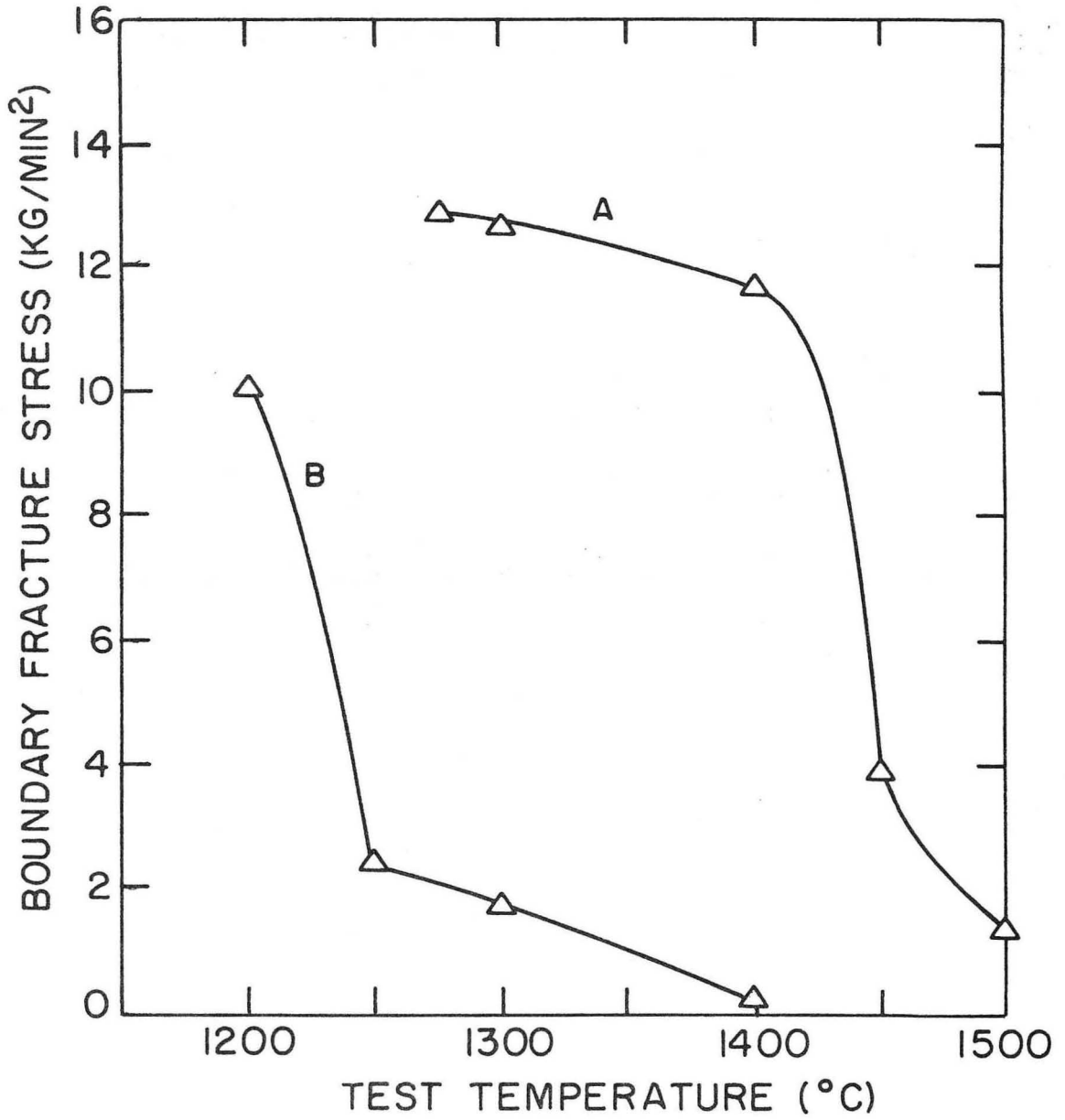
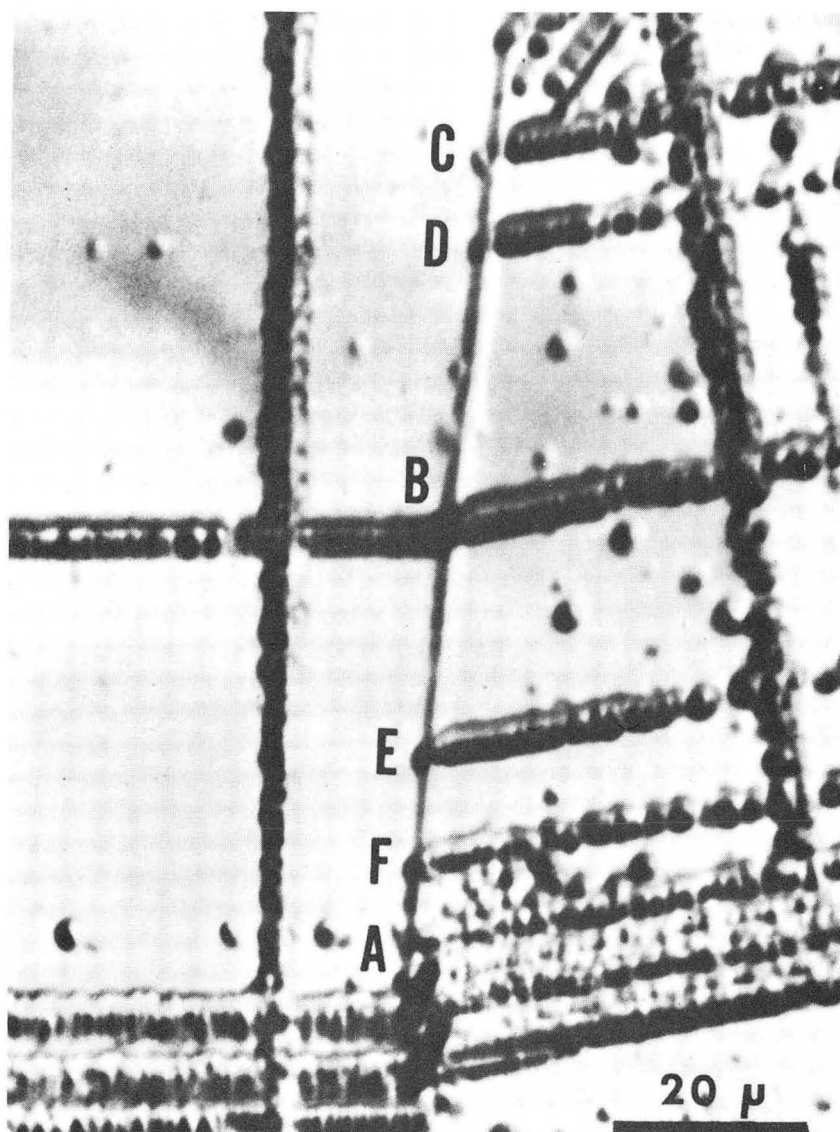


Fig. 20

XBL 686-1115



XBB 686-3951

Fig. 21

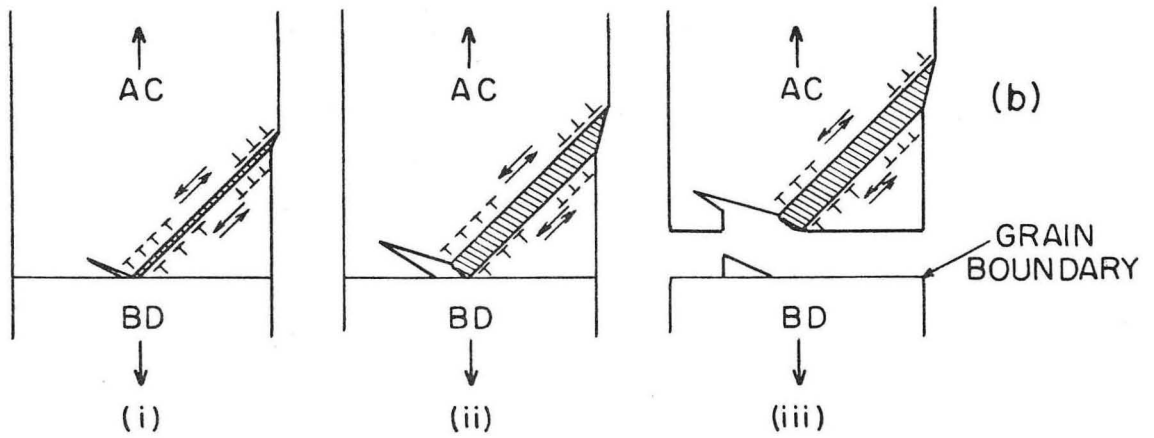
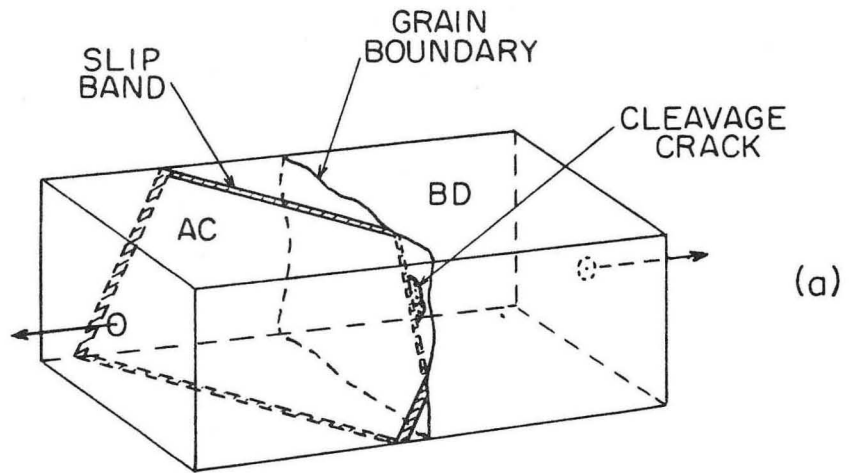
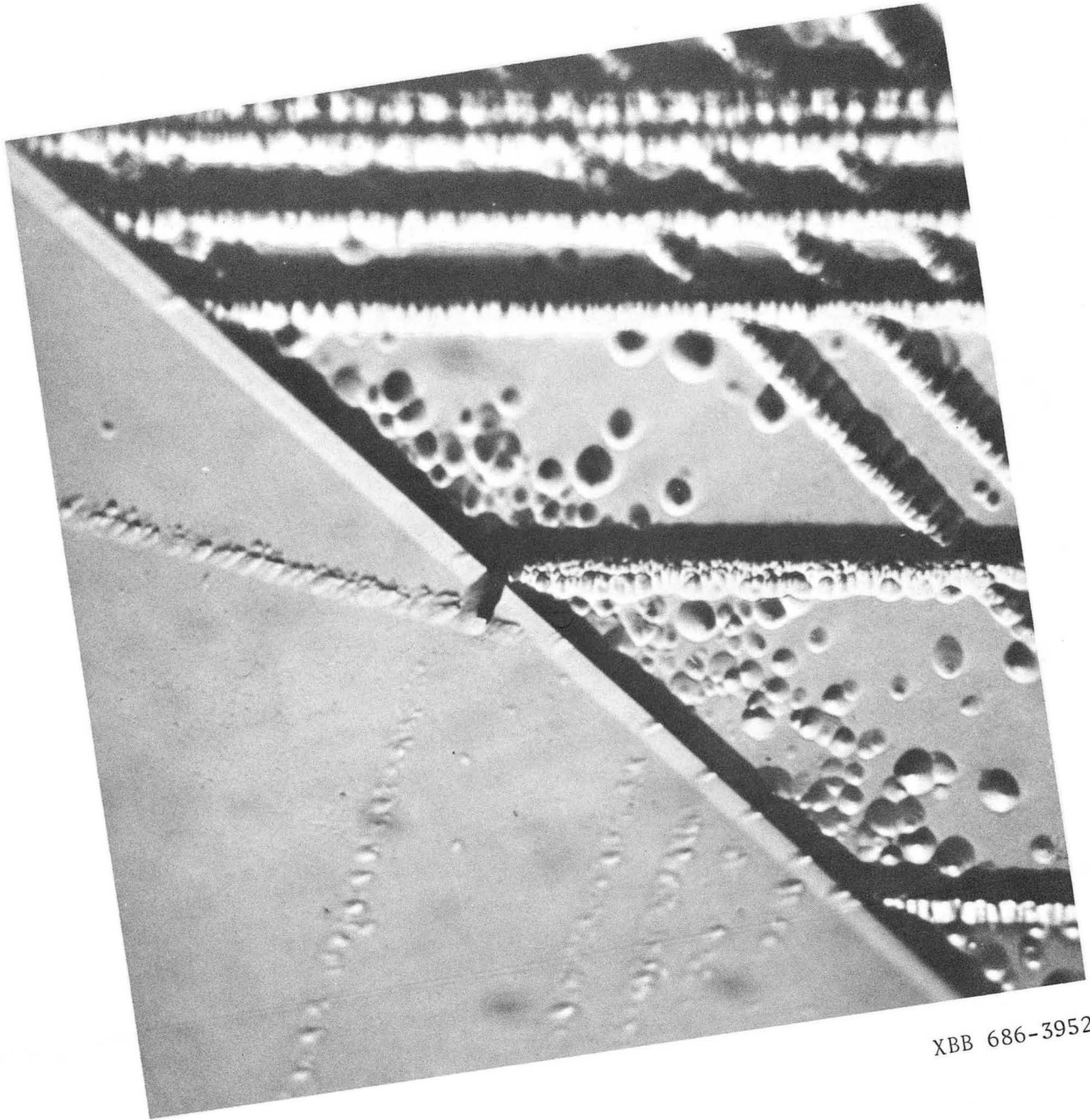


Fig. 22

XBL 686-1116



XBB 686-3952

Fig. 23

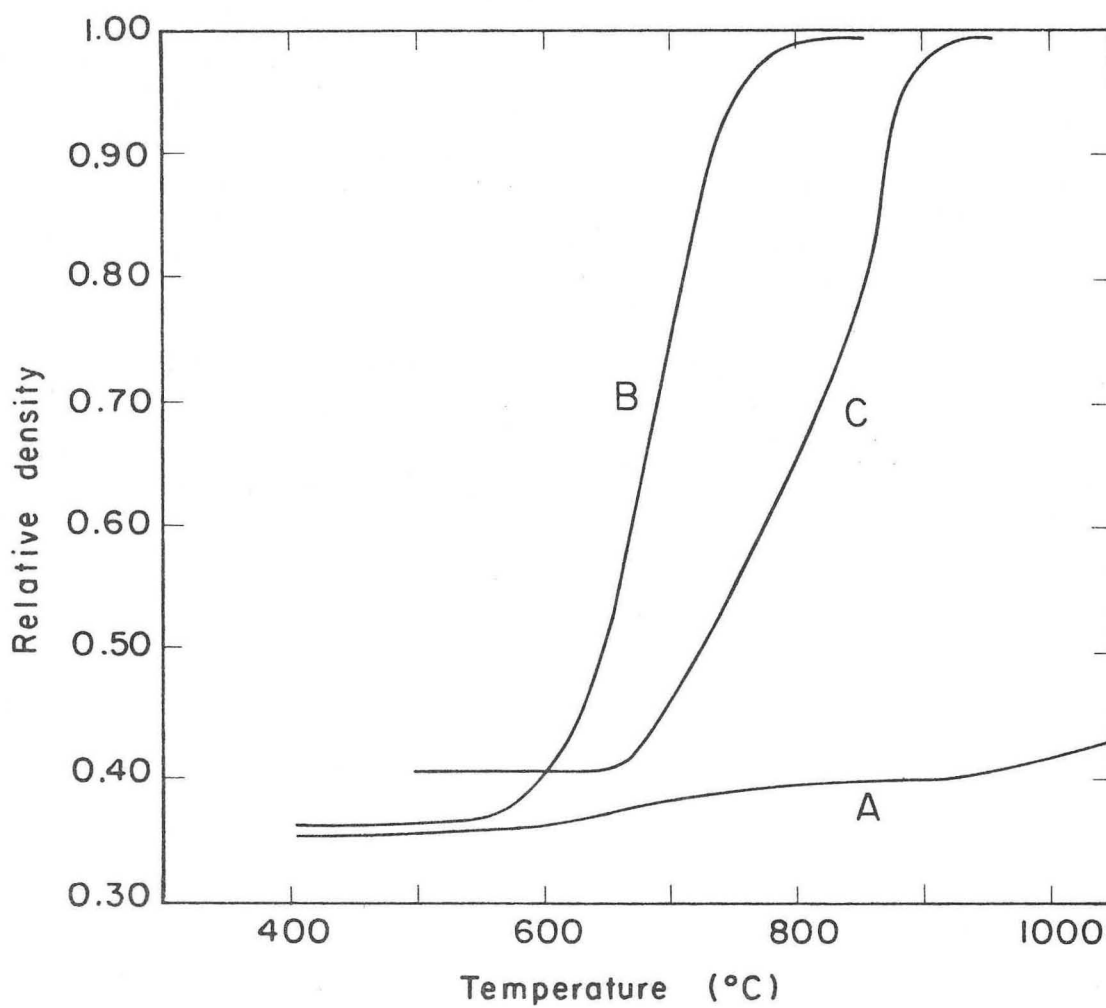
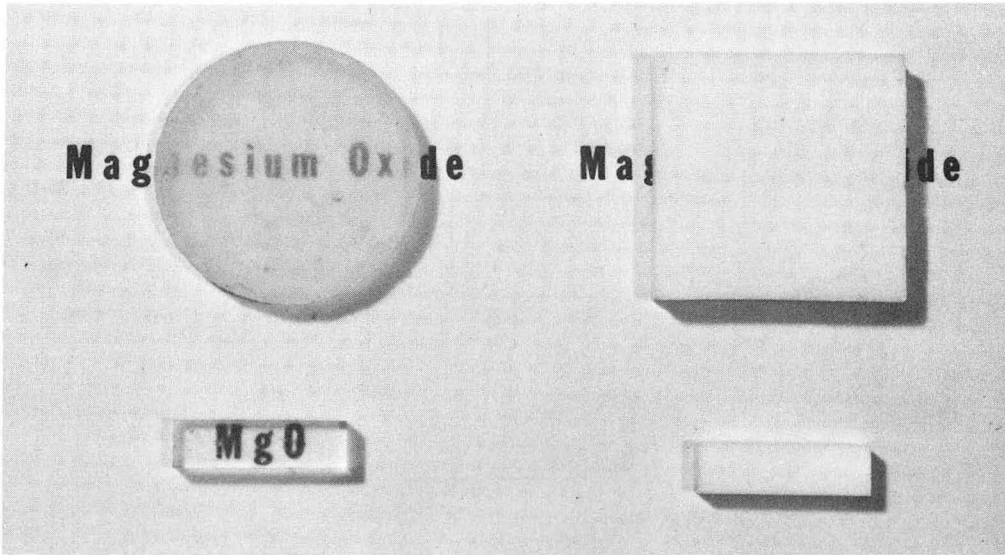
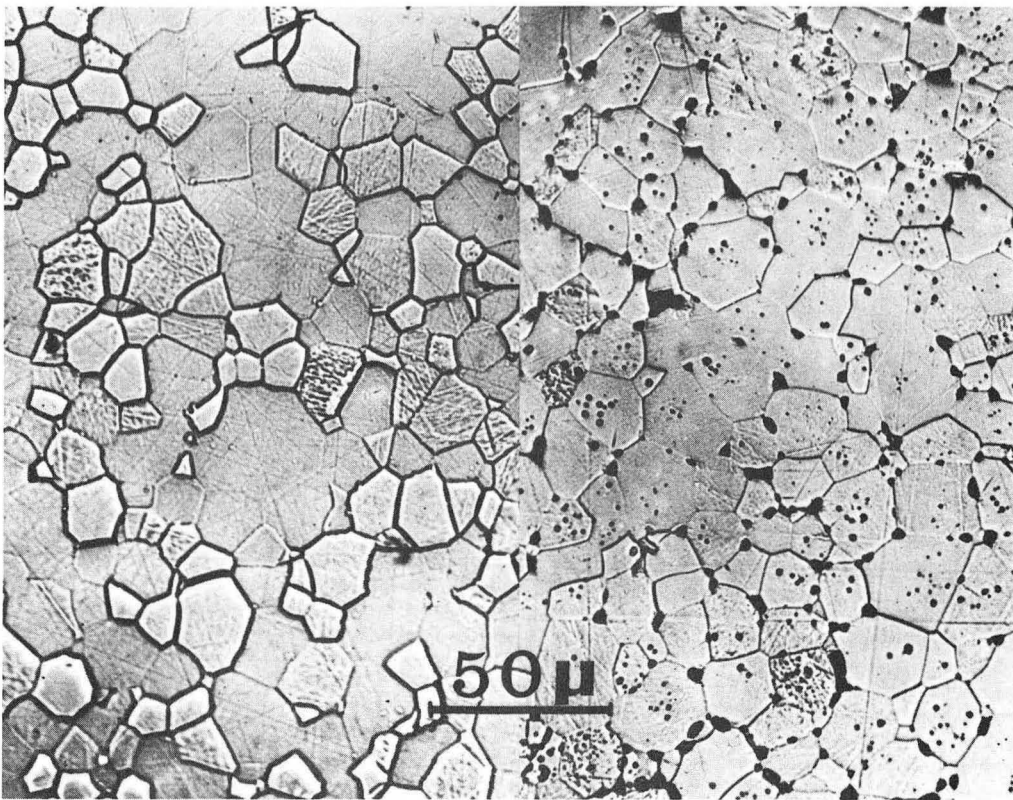


Fig. 24

MUB 11923



ZN-6010



ZN-6011

Fig. 25

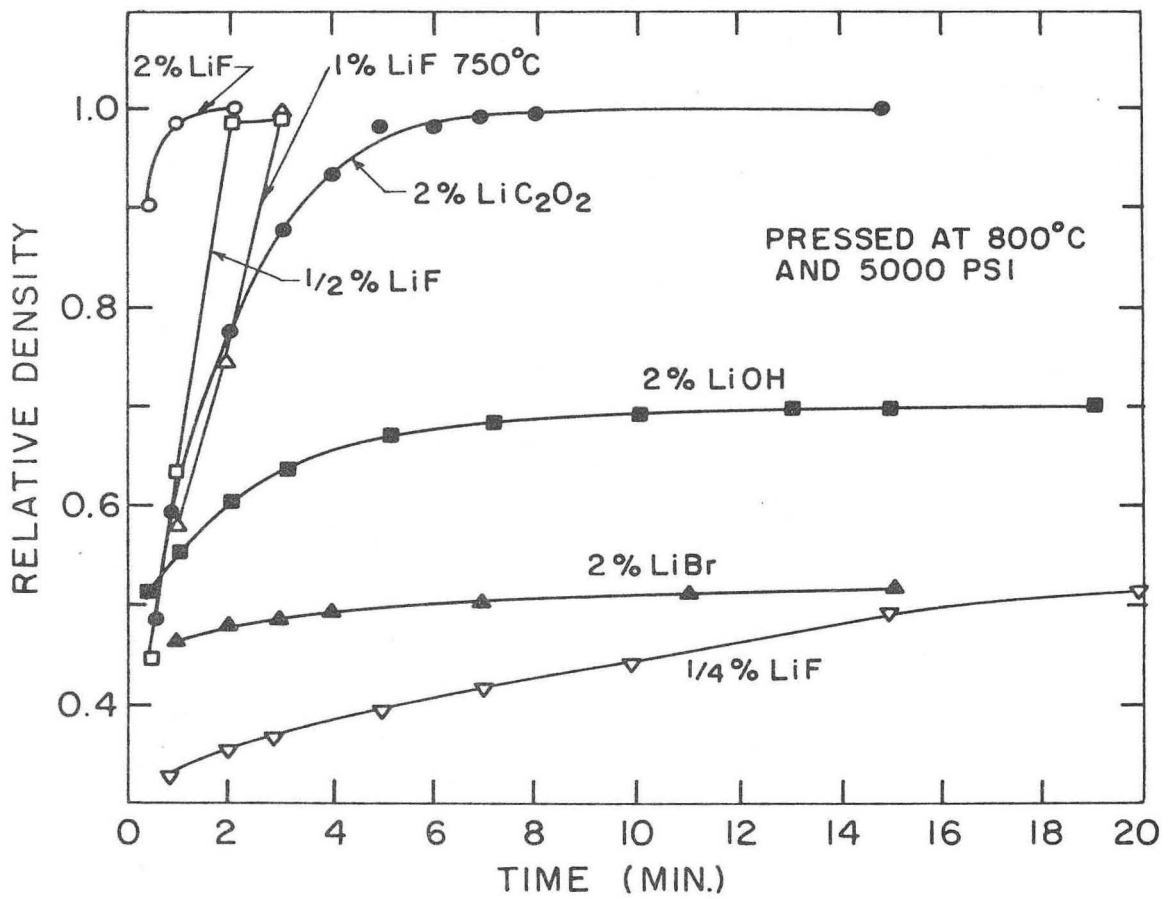
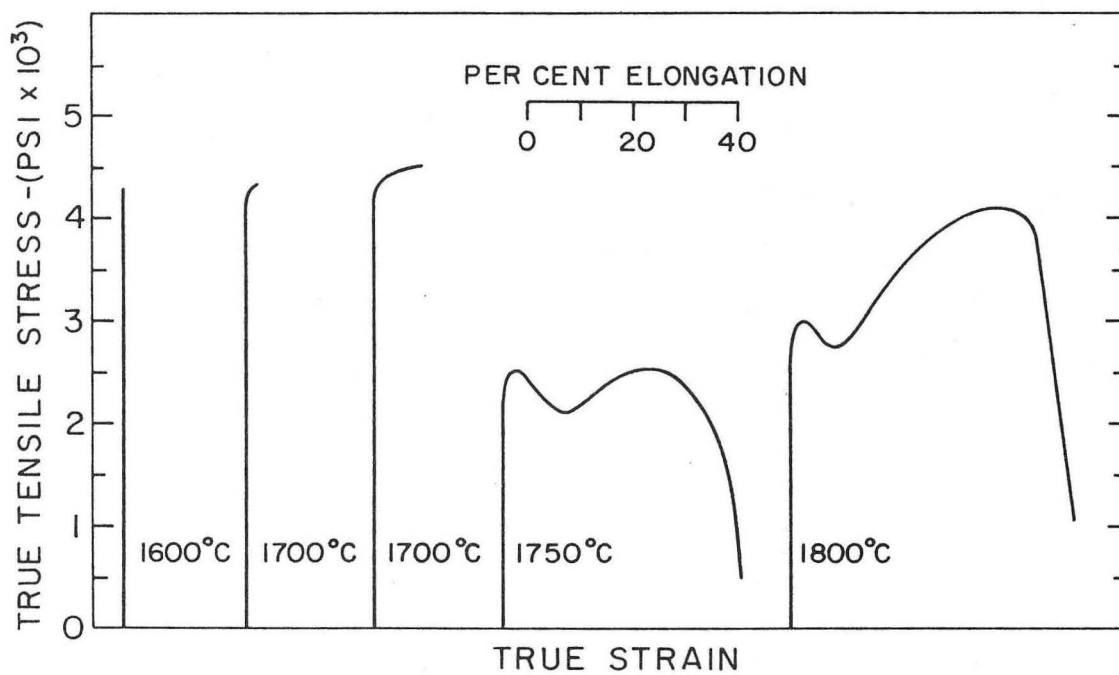


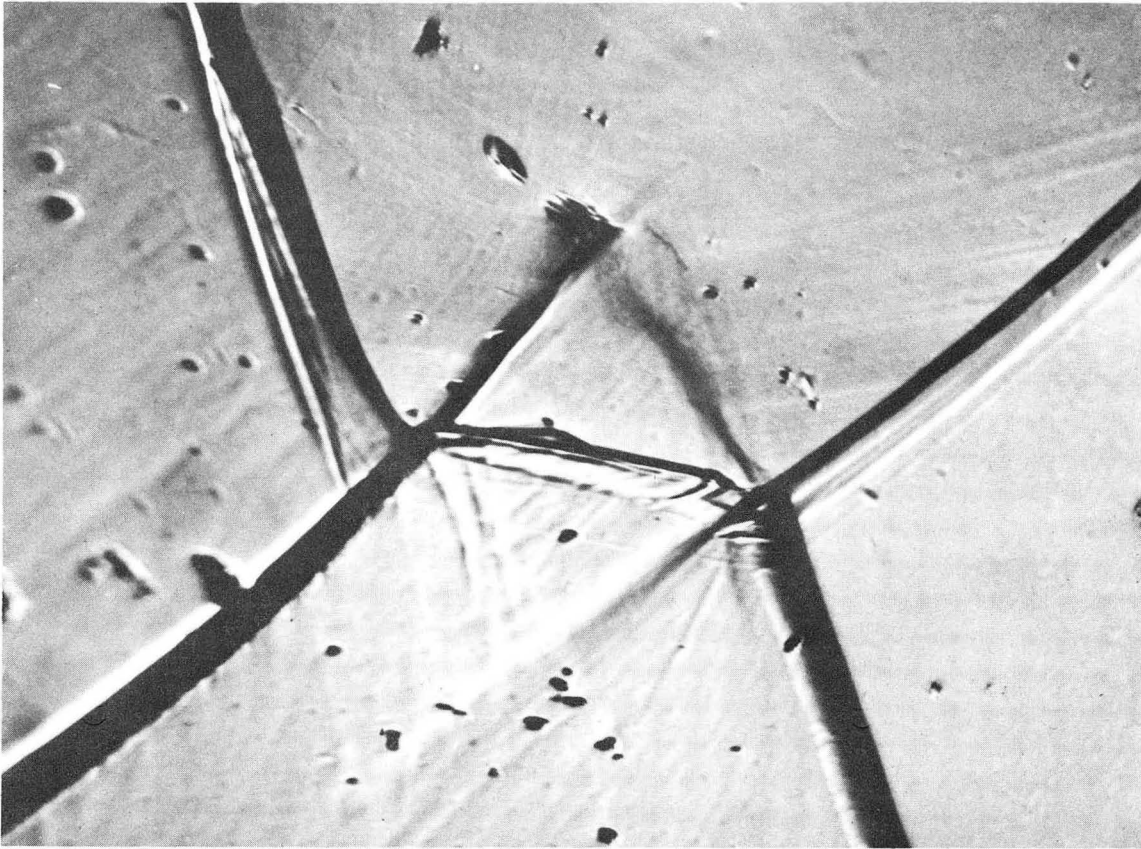
Fig. 26

XBL 686-1117



XBL 686-1118

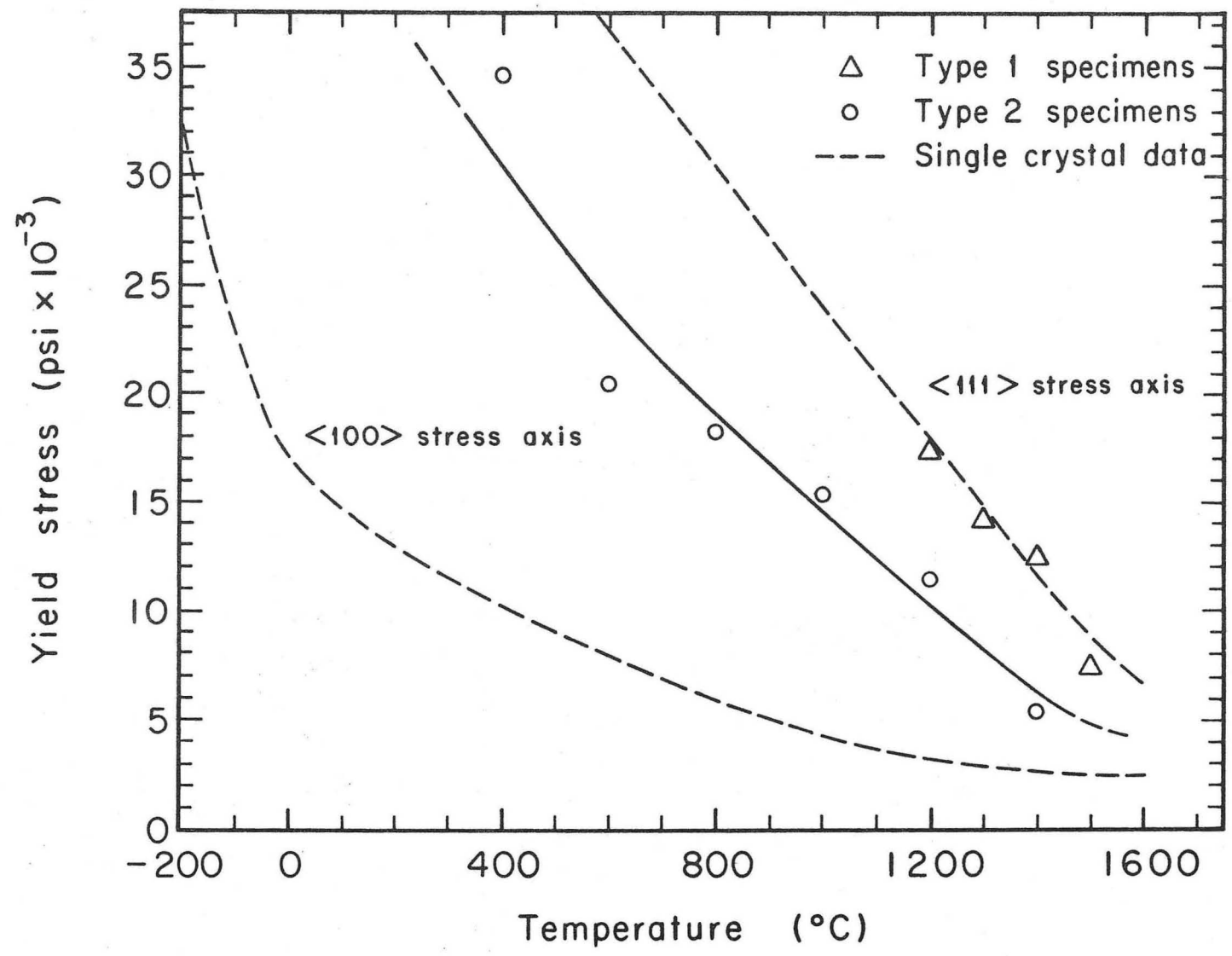
Fig. 27



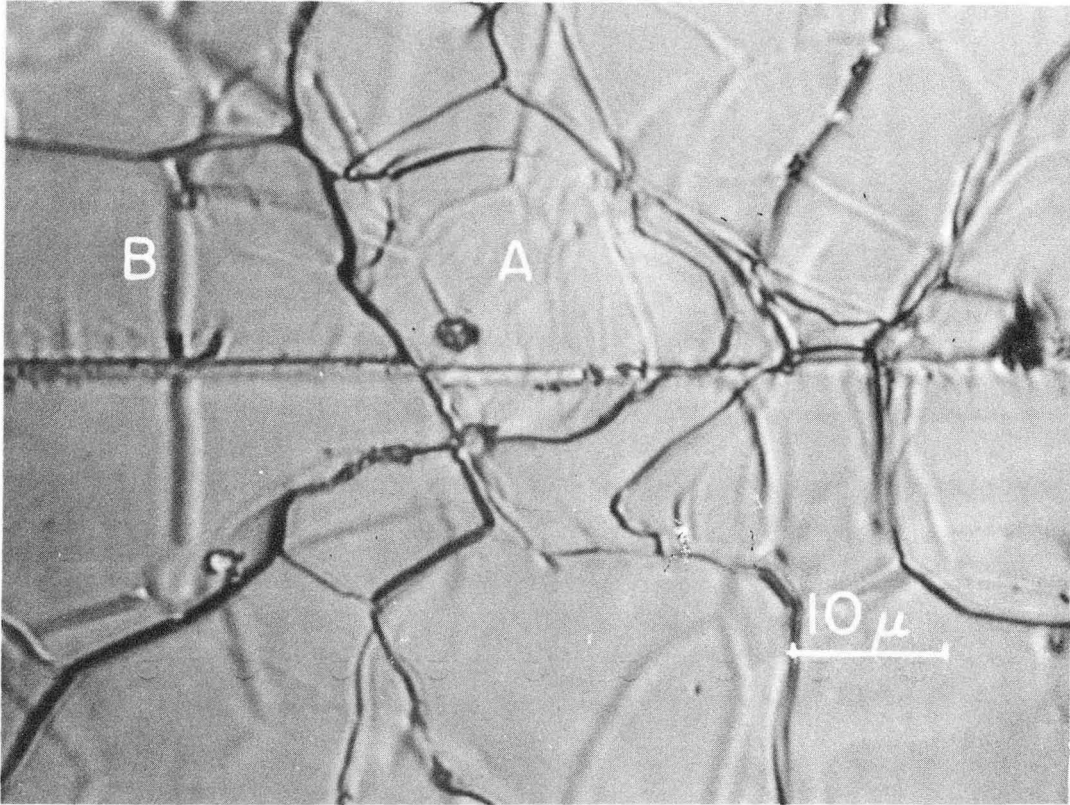
XBB 686-3953

Fig. 28

Fig. 29

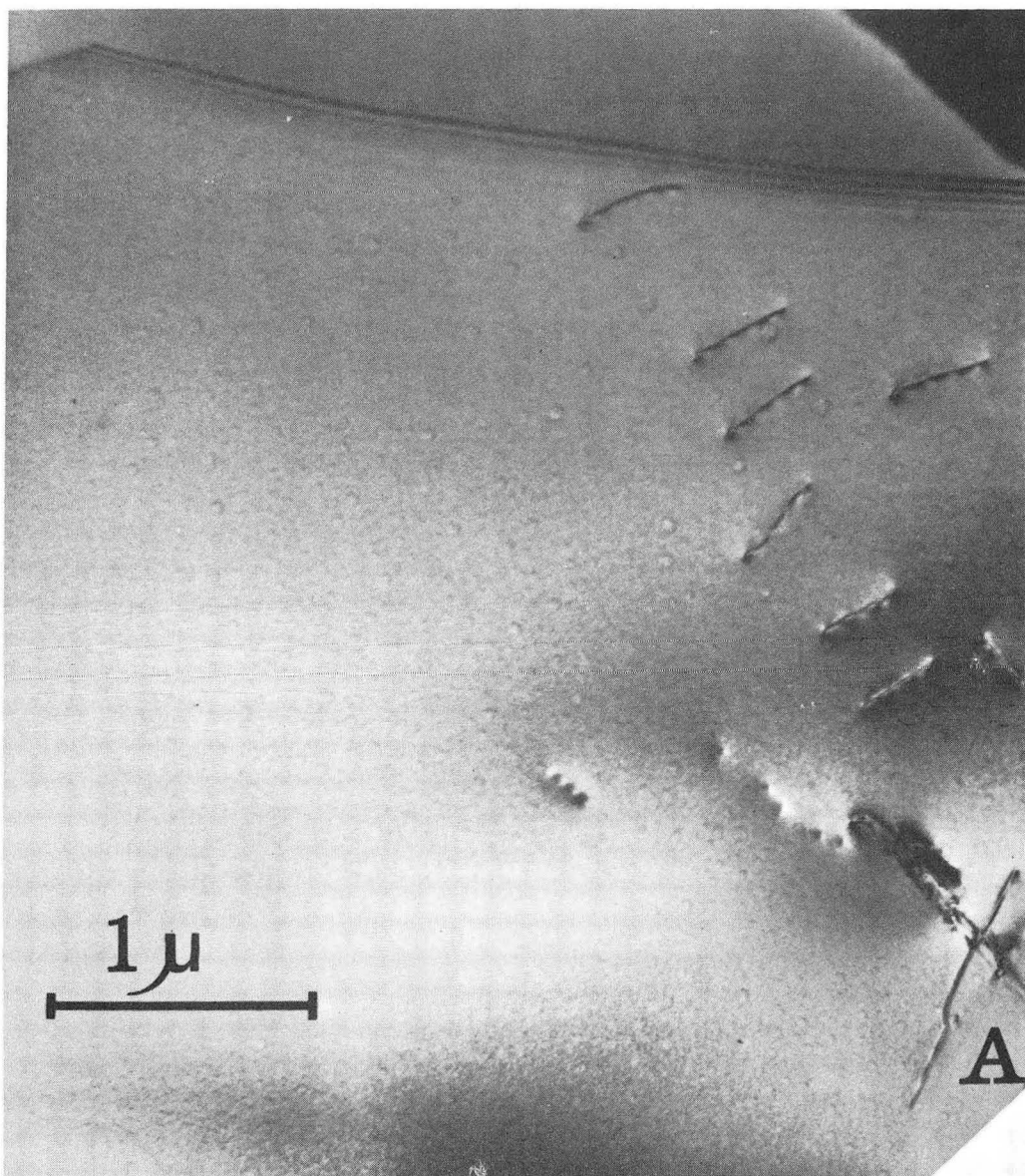


MUB-4377



ZN-4707

Fig. 30

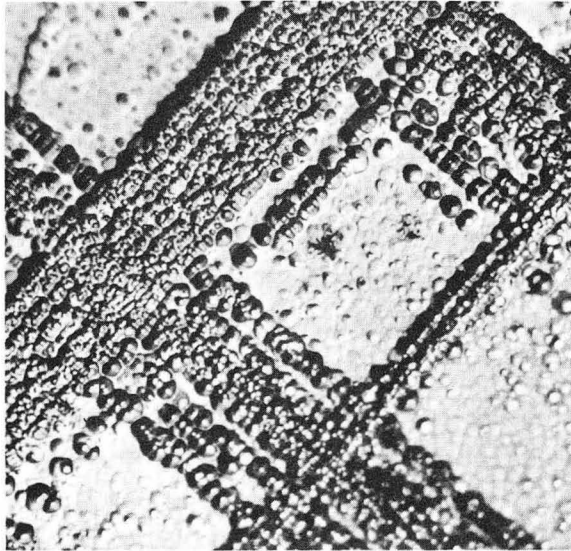
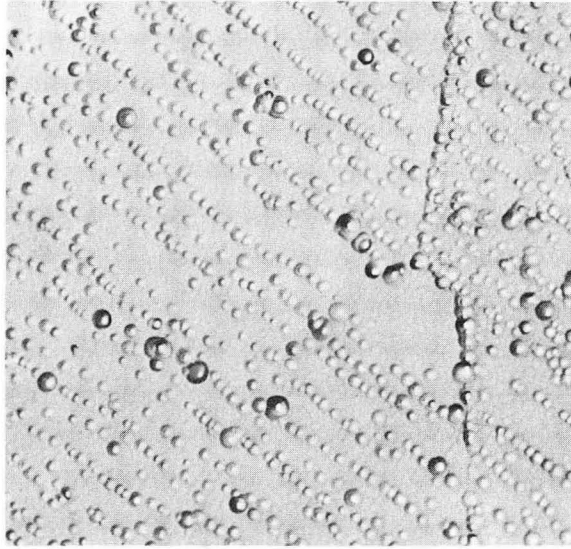


ZN-5922

Fig. 31

Fig. 32

XBB 686-3949



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