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FRACTURE PROCESSES IN GERMANIUM

TECHNICAL DOCUMENTARY REPORT NO. ASD-TDR-62-1029

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Directorate of Materials and Processes
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

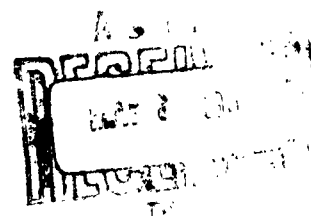
Project No. 7350, Task No. 735001

(Prepared under Contract No. AF 33(616)-6832
by the University of Utah, Salt Lake City, Utah;
O. W. Johnson, N. Farb, and P. Gibbs, authors.)

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Unclassified Report

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The effect of atmosphere on plastic deformation of both single crystal and polycrystalline Al_2O_3 was examined. Heat treatment in dry O_2 and N_2 at temperatures between 1350° and 1580°C for periods up to 50 hours, produced no significant changes in creep behavior.

1. Germanium
2. Aluminum Oxide
3. Fracture
4. Creep
- I. AFSC Project 7350, Task 735001
- II. Contract AF 33(616)-6832

- III. University of Utah, Salt Lake City, Utah
- IV. O. V. Johnson, M. Barb. P. Gibbs
- V. Avail fr OTS
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FOREWORD

This report was prepared by the Solid State Physics Department of the University of Utah under USAF Contract No. AF 33(616)-6832. This contract was initiated under Project No. 7350, "Refractory Inorganic Non-metallic Materials," Task No. 735001, "Refractory Inorganic Non-metallic Materials: Non-Graphitic." The work was administered under the direction of the Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio. Mr. John D. Latva and, subsequently, Mr. J. B. Blandford, Jr. acted as project engineers.

This report covers work done from 1 June 1961 to 30 November 1962.

ABSTRACT

The fracture strength, σ_F , of germanium, at constant loading rate has been determined over a temperature range from -196°C to 500°C . σ_F decreases slowly up to about 200°C , then increases to about 400°C , followed by subsequent decrease. σ_F is found to be essentially independent of ambient for a wide variety of chemical species. These observations are analyzed in terms of fracture nucleation at a flaw near, but not exposed to, the surface, by a presently unknown kinetic process. The role of dislocations apparently is to inhibit fracture by relaxation of stress concentrations up to about 400°C , above which general plastic deformation precedes fracture, suggesting the possibility of fracture nucleation by dislocation pileups. The applicability of this analysis to another brittle system, Al_2O_3 , is considered in the light of its reported fracture, deformation, wear and friction characteristics. Striking similarities are found in the behavior of these two crystals, suggesting that the underlying mechanisms must have much in common.

The effect of atmosphere on plastic deformation of both single crystal and polycrystalline Al_2O_3 was examined. Heat treatment in dry O_2 and N_2 at temperatures between 1350° and 1580°C for periods up to 50 hours, produced no significant changes in creep behavior.

This report has been reviewed and is approved.



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FRACTURE PROCESSES

PART A: FRACTURE PROCESSES IN GERMANIUM

I. Introduction:

Although considerable work has been done on the low temperature deformation and fracture characteristics of germanium and silicon,¹⁻⁸ no substantial agreement has yet been achieved, and in fact the literature contains a variety of conflicting reports. In particular, the relative importance of such factors as mechanical condition of the crystal surface and the chemical environment has not been clearly established.^{1,2,3,5} The generation and motion of dislocations in these materials at low temperatures (less than 350°C for germanium and 450°C for silicon) has not been unambiguously demonstrated and their role in the fracture process is currently open to question.

The authors have recently described experiments^{9,10} on germanium single crystals which appear to establish the following points: (1) "Fracture strength" varies reproducibly from about 10 kg/mm² for specimens lapped with medium grit abrasive to an average of 300 kg/mm² or more for carefully etched specimens, with intermediate values for specimens lapped with finer abrasive or for specimens etched to a depth less than the penetration of the surface damage. (2) These strengths depend only on the mechanical condition of the surface, and not on the presence of an etching solution. (3) The depth of penetration of the damage produced in preparation or handling is almost an order of magnitude greater than predicted by the Griffith equation

$$\sigma_c \approx \left(\frac{E\gamma}{l} \right)^{1/2} \quad (1)$$

where σ_c is the stress at fracture, E is Young's modulus, γ is the surface energy and l is the crack length. Such surface damage is invariably produced by any contact with harder material.* (4) Germanium is subject to "static fatigue." That is, a specimen stressed at a constant level near the expected fracture stress will often fracture after a time delay, which typically is of the order of a few seconds, but has been observed to range as high as 30 hours at room temperature.

The work reported herein is a continuation of that described above and is part of an overall study of fracture phenomena in these crystals. The current paper deals primarily with the points of confusion previously mentioned. In Section II, the dependence of fracture strength on temperature and the apparent relationship of changes in strength to changes in dislocation mobility are described. Tests of fracture strength in a variety of chemical ambients are described

* A variety of experimental evidence was recently reviewed by Buck,¹¹ all of which indicated a penetration of surface damage deeper than predicted by Equation (1), but somewhat more shallow than the penetration indicated by fracture studies.^{9,10}

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in Section III, and the implications of these results are discussed in Section IV. Analysis of these results has led to formulation of a phenomenological theory of fracture, which permits classification of a wide variety of fracture-related processes on a fundamentally consistent basis, and which suggests a new philosophy of experimental study of fracture. The authors feel this may represent a substantial contribution. The material is presently being prepared for publication, but time did not permit inclusion in this report.

II. Effect of Temperature on Fracture:

Figure 1 shows values of "fracture strength" σ_F (maximum outer fiber tensile stress at the time of fracture, calculated from the simple beam equation) for germanium specimens at various temperatures from -196°C to 500°C . The calculated value of σ_F at 500°C is probably somewhat high, since a significant amount of plastic deformation occurred prior to fracture. The specimens were prepared by cutting with a diamond saw, then lapping at least 75 microns from each surface with 1200 grit abrasive. Part of the specimens were subsequently lapped with 3200 grit abrasive. This resulted in an increase in fracture strength of about 15 per cent. No other changes in fracture behavior were detected. The values indicated in Figure 1 are the "normalized" fracture strengths, $\sigma_F(T)/\sigma_F(25^\circ)$, thus eliminating the effect of this difference in preparation. Specimens were broken in three-point bending with an average loading rate of about $0.4 \text{ kg/mm}^2 \text{ sec}$. ("Step-function" loading was used, with increments of 1.5 kg/mm^2). Figure 1 indicates a gradual decrease in fracture strength with increasing temperature, up to approximately 200°C , after which σ_F increases quite sharply to about 400°C then rapidly decreases as the temperature is raised further. These temperature ranges will hereafter be referred to as Region I, II, and III respectively. Similar results, obtained under different experimental conditions, have recently been reported by Suzuki and Kojima¹².

Figure 2 shows the fracture surface of a specimen broken at 300°C , after etching lightly in a dislocation etchant (referred to as "DE-2" in Reference 10). Fracture initiated at the focal point of the cleavage steps, on (or near) the tensile surface of the specimen. The rows of pits around this "fracture nucleus" apparently represent dislocations which were produced prior to fracture. They are not seen after fracture at lower temperature. However, similar arrays were found in specimens prestressed at 300°C , then fractured at a lower temperature. The specimen of Figure 2 was loaded at a rate somewhat slower than those represented in Figure 1, but similar though less extensive arrays of dislocation pits were found on 300°C specimens in Figure 1.

Figure 3a shows the fracture surface of one of the 400°C specimens of Figure 1. The rows of pits near the fracture nucleus again presumably represent dislocations generated prior to fracture. The more dense arrays of pits in the rounded areas further from the nucleus are imperfection arrays produced during fracture and apparently have no bearing on the initiation process. These dense arrays are confined primarily to a thin layer on the fracture surface, as

can be seen by comparison of Figure 3a with Figure 3b, which is the same crystal after approximately 6 microns has been removed from the surface. These arrays of etchable imperfections are described in detail elsewhere.^{9,10}

Figure 4 shows one of the 500°C specimens represented in Figure 1. Extensive plastic deformation obviously preceeded fracture in this case.

Fracture strength is sensitive to loading rate, at almost all temperatures. In Regions I and III, an increase in loading rate generally results in increased fracture strength, but this relationship is reversed in at least the lower part of Region II. The dependence on loading rate appears to be quite complicated, however, and these results are probably valid only for the particular test conditions used. For example, pre-straining of specimens at 325°C at a constant level of 20 kg/mm² for 15 min. results in an increase of about 50 per cent in σ_F , for specimens broken at the usual loading rate (0.4 kg/mm² sec.). However, an increase by a factor of 6 in the loading rate at 325°C produced no statistically significant change in σ_F (actually, there appeared to be a small increase in σ_F).

III. Effect of Chemical Environment on Fracture:

Because of the reported^{2,5} influence of acid environments on fracture strength of germanium, the most extensive tests were made in HF and in varying proportions of HF and HNO₃. All tests were made at room temperature; all other test conditions were similar to those described above.

Figure 5 shows the results obtained for lapped specimens broken in HF, compared to identical specimens broken in air. The average stress at fracture was 27.5 kg/mm² and 28.3 kg/mm² respectively. This difference is clearly not statistically significant. Using standard statistical methods, it was determined that these data indicate a probability of < 1% of a difference in fracture strength of as much as 20%. A difference of as much as 10% carries a probability of approximately 20%.

Tests were also made of etched samples in varying proportions of concentrated nitric and hydrofluoric acids. No attempt was made to measure possible effects of these acids on the strength of lapped specimens. The tests of lapped specimens in HF described above were possible because HF does not etch germanium at an appreciable rate. However, mixtures of HF and HNO₃ attack germanium vigorously, and because of the extreme sensitivity of fracture strength to the mechanical condition of the surface (discussed in Section I above), it did not appear feasible to attempt to separate any possible environmental effect from the effect due to etching of the surface flaws. The data obtained are presented in Figure 6. The specimens were cut and lapped together, then etched to remove at least 75 microns from each surface. They were broken in approximately 15 seconds under constantly increasing stress, to avoid excessive reduction in cross section due to etching. The control series (in air) represents 15 specimens; the other series consist of 5 specimens each. The mean deviation for the control samples was 72 kg/mm²; this value is typical of etched samples and accounts for the rather large uncertainty limits. Analysis of the data in Figure 6 indicates that the differences among the series are not statistically

significant, except for a questionable significance (uncertainty 2%) between the 100% HNO_3 , 95/5% HNO_3/HF series. It should be noted that the mean value of σ_p in air is less than in HF. While the difference is not significant in a statistical sense, the data do indicate the improbability of a weakening in HF. On the basis of these data, a reduction in strength of 20% in HF carries a probability of less than 5%, in contrast to the reduction in strength by a factor of 14 previously reported.⁵

In addition, less extensive and detailed tests were made in a variety of other media. Both etched and lapped samples were tested in distilled water, in ethyl alcohol, acetone, sodium hydroxide (2 N), sulphuric, hydrochloric and acetic acids in various concentrations up to 10% (none of these acids etch germanium at a measurable rate), and in a variety of mineral oils. In none of these media was any change of the fracture strength observed. Even though as few as six specimens were broken in some cases, a significant change almost certainly would have been detected. Tests of the effect of exposure to air of both lapped and etched specimens for periods up to four months were also negative.

IV. Conclusions:

On the basis of the observations described above, it seems reasonable to draw the following tentative conclusions:

1. Dislocation generation and/or motion is apparently not involved in fracture initiation in Region I. The enhancement of strength in Region II coincides with the onset of plastic deformation, as revealed by the etching studies, and is probably due to relaxation of the stresses around potential fracture sources. The onset of plastic deformation in Region II would also account for the change in the sense of the dependence of fracture strength on loading rate between Regions I and II, by allowing relaxation of stress concentrations.

Recent measurements¹³ of the temperature dependence of dislocation velocity at elevated temperatures yielded an activation energy of about 1.6 eV. It is not yet certain what mechanism limits the motion of dislocations in covalent crystals. However, if the mechanism is the same at room temperature as in the higher temperature range, some information about the processes involved can be obtained from simplified calculations of the stress required to move a dislocation independent of temperature. The force on a length l of dislocation due to a resolved shear stress τ is

$$F = \tau lb \quad (2)$$

where b is the Burger's vector of the dislocation. If the "activation length" l is one interatomic distance along the dislocation, then the force impeding dislocation motion almost certainly is the Peierls force. That this is probably the case may be seen by assuming the Peierls force F_p to be sinusoidal with period (b) , then equating the activation energy E (1.6 eV) to the integral of F_p to the "top of the barrier":

$$E = \int_0^{b/2} F_p dx = \frac{F_{p(max)} b}{\pi} \quad (3)$$

The numerical value of $F_{p(max)}$ from Equation (3) now may be used in Equation (2) to obtain an estimate of the maximum shear stress which may be applied to a crystal without temperature-independent (and catastrophic) dislocation motion. The value of τ obtained in this way for germanium is 1250 kg/mm², which is about half the theoretical strength. Similar order of magnitude calculations suggest that thermal fluctuations would reduce this to about 800 kg/mm² at room temperature. Since carefully etched germanium crystals have been observed⁹ to withstand stresses of nearly 400 kg/mm² for considerable periods, with no indication of dislocation motion, the interpretation of the factor limiting dislocation motion as being the Peierl's force is probably correct. This is particularly reasonable in the case of germanium because of the nature of the bonding. If these conclusions are essentially correct, it is reasonable to expect plastic flow to occur at low temperatures only in localized regions near stress concentrators, such as micro-cracks. The size of the region in which flow could occur would increase with temperature, resulting in more effective stress relaxation at higher temperatures, thus accounting for the transition from Region I to Region II in a natural, consistent manner.

The cause of the transition from Region II to Region III is a matter of speculation at present. However, Figure 4 shows that a substantial amount of plastic deformation occurred prior to fracture in Region III, and it seems likely that one or more of the several mechanisms of fracture nucleation by dislocation interaction which have been proposed¹⁴⁻²¹ may operate in this region. It should, of course, be recognized that the temperature ranges of these three regions very likely depend on loading rate, and that the remarks above and those to follow refer only to crystals fractured at the loading rates specified.

2. Observation of a decrease of fracture strength with increasing temperature in Region I, and also the fact that germanium is subject to static fatigue,⁹ appear to require that a thermally activated process, resulting in the enhancement of pre-existing surface defects, precede fracture.

3. The apparent absence of dependence of fracture strength on chemical environment at the time of fracture suggests that the initiating defect is not chemically exposed to the exterior until after the critical stage has been passed. This observation, and the discrepancy between the observed depth of surface damage and that predicted by the Griffith equation (Equation 1), appear definitely to rule out a simple Griffith mechanism of fracture.

It is suggested that an interpretation of these data along the following lines appears to be the most plausible at present: High local stresses produced in cutting or handling the specimen result in opening small cracks in the surface. The stress distribution would be such as to preclude propagation of these cracks more than a short distance, and since the stresses would be of a transient nature, the crack would reclose and the material would be expected to "heal" to some extent. This process would result in a flaw in the surface (such as that referred to by J. W. Allen²² as a "dislocation crack") which could be described as a planar array of imperfections and impurities. Misregistry of the crack

surfaces would result in a "wall" of concentric dislocation half-loops. Large numbers of point imperfections would probably also be created.

Such a defect would be expected to exhibit some strength, but would certainly be weaker than bulk material. A further weakening of such a flaw could result from a number of possible processes which might occur within this defect, under the influence of applied stress, such as growth of voids by vacancy migration (either from vacancies produced during the cracking process, or from dislocation climb), coalescence or rearrangement of dislocations by either climb or glide (or both) or perhaps growth of particles of a second phase, resulting from migration of trapped impurities. This interpretation is consistent with observations of the damage arrays occurring on fracture faces (see Figures 3a and 3b). These arrays have all of the etching characteristics which would be expected of flaws produced in the manner described above, and their occurrence is confined to regions where one might expect very large local stresses, such as would be required to produce forking of the propagating cleavage crack.

It seems likely that much of the confusion relative to the influence of chemical environment and aging on fracture strength may have resulted from experimental errors due to the extreme sensitivity of germanium to surface damage and to the unexpectedly large penetration of this damage, neither of which has been generally recognized.

PART B: APPLICABILITY OF RESULTS TO OTHER MATERIALS

I. Review of Experimental Results:

It seems worth while at this point to explore the possibility that the brittle characteristics of other materials may be accounted for, at least qualitatively, by mechanisms similar to those suggested in the previous section for germanium. In particular, although it's crystalline structure and bonding are drastically different from germanium, aluminum oxide appears to have macroscopic mechanical properties somewhat similar to those of the elemental semiconductors.

Wachtman and Maxwell²³ measured the rupture strength of sapphire rods in the temperature range from room temperature to 1100°C, for rods with the C-axis parallel to the rod axis, and rods with C-axis inclined at 45° to the rod axis (see Figure 7). The loading rate was about 12 kg/mm² min., after an initial load of 10 kg/mm². In both cases, they found a distinct minimum in the fracture strength at roughly 600°C, although the minimum was more pronounced for the 45° rods.

More recent work by Charles²⁴ verified the general features reported above, although it was also shown that a stress-corrosion mechanism (probably due to water vapor) may significantly reduce the fracture strength of both single crystals and polycrystalline Al₂O₃. This effect was probably minimized in the work of Wachtman and Maxwell by their fairly rapid loading rate. Charles reports an orientation dependence in air at room temperature (strain rate unspecified) essentially identical to that found by Wachtman and Maxwell, for specimens broken in 4-point bending. Values approximately 50% higher were obtained at liquid nitrogen temperatures. The temperature dependence of fracture stress (maximum tensile stress at fracture, four-point bending) for 60° rods broken in vacuum is indicated in Figure 7. Similar tests were performed on polycrystalline specimens (Figure 8). Results are qualitatively similar to those obtained for single crystals up to 600°C. An apparent decrease in the dependence of fracture strength (in single crystals) on loading rate with increasing temperatures between -35°C and 200°C was also reported.

Wachtman and Maxwell's²³ work on 45° rods suggested a decrease in fracture strength above 1100°C (see Figure 7). This decrease was verified in recent tests by Stofel and Conrad²⁵ on 60° rods broken in tension between 1100° and 1500°C. These authors found the fracture strength correlated quite well with total plastic strain, over a range of strains from 10⁻⁴ to 0.6. Fracture strength, at a given strain rate, was found to decrease as temperature was increased, although the correlation was not as good as that with total strain. Further evidence of the importance of plastic deformation was inferred from the point of initiation of fracture, which was at the point on the surface corresponding to the longest path-length for gliding basal dislocations in 90% of the crystals examined. Values of strength reported by these authors are generally somewhat smaller than those previously discussed for similar experimental conditions. This presumably reflects a difference in the surface preparation.

Brenner²⁶ studied the fracture strength of sapphire whiskers in tension, from room temperature to the melting point. Specimens were broken in air, but

the loading time was quite short (less than 10 seconds), thus minimizing the effects of stress corrosion mechanisms. Fracture strength was found to decrease from an average of 640 kg/mm² at room temperature to 190 kg/mm² at 1550°C, after which it decreased more slowly up to temperatures near the melting temperature. No measurements of deformation prior to fracture were made.

From the results summarized above, it seems clear that a successful explanation of the fracture behavior of sapphire must take account of its plastic deformation properties. Bridgeman²⁷ reported massive deformation, involving both slip and twinning, under hydrostatic pressure of 20,000 and 30,000 atmospheres and compressive stresses of 400 to 850 kg/mm² at room temperature, with strong orientation dependence. The present authors are not aware of any reports of macroscopic plastic deformation at atmospheric pressure below about 900°C. Two slip systems have been shown to operate in sapphire. Slip on the basal system occurs readily above 900° or 1000°C.^{28,29} Slip on the prismatic system is known to occur²⁹ above 1600°C. Twinning has been observed^{25,27,30} only under compressive stresses, either under hydrostatic pressure with large applied stresses at room temperature, or at temperatures of 1100°C or more under atmospheric pressure.

In many cases, wear and surface friction appear to involve the same or similar processes to those responsible for plastic deformation and fracture. Steijn³¹ studied the wear of sapphire on tungsten carbide as a function of orientation and rubbing direction at room temperature. The greatest wear resistance (and the smoothest resulting surfaces) was obtained when the orientation and rubbing direction were most favorable for plastic flow. A very rapid, brittle type of wear was observed for rubbing in the direction of the C-axis on a prismatic face. Neither of the known slip systems would be expected to operate to a significant extent under these circumstances. The author reported evidence, from etching studies, for the penetration of some type of damage to a depth greater than 25 microns. The author suggests that this damage may consist of dislocations produced by plastic deformation of the surface layer. Further evidence for plastic deformation cited by the author is the apparently amorphous nature of the wear debris, as determined by both x-ray and electron diffraction techniques.

Riesz and Weber³² and previously Riesz,³³ studied the friction of sapphire on sapphire over the temperature range 25° to 1550°C. In atmosphere, the coefficient of friction decreases with increasing temperature to a pronounced minimum at about 600°C. The authors attribute this to the action of a surface film which is desorbed at about 600°C. In vacuum, generally higher coefficients of friction are observed. Below about 300°C, a "slip-stick" type of friction is usually observed, accompanied by sub-surface fracture in some cases. Friction decreases in the 300° to 1000°C range, accompanied by a change from "slip-stick" to smooth, sliding friction. Friction is observed to increase again above 1000°C. The authors report etching studies which indicate production of basal dislocation at 600°C.

II. Discussion:

The experimental results discussed above show a very close parallel between the fracture behavior of Al₂O₃ and germanium. In particular, both materials

exhibit three distinct regions, in a plot of fracture strength vs temperature at constant loading rate. In both cases the fracture strength decreases with increasing temperature in Region I, increases in Region II and falls off again in Region III. The transition temperatures, expressed as fractions of the melting temperatures, are the same within experimental error (0.4 and 0.6).

Charles²⁴ has suggested that the decrease in strength at low temperatures may be due to changes in the elastic modulus. While there is a close similarity in the behavior of the elastic modulus and the fracture strength of polycrystals above 600°C (Figure 8), and these two quantities may well be related in this temperature range, this explanation does not seem adequate to account for the observed 20% decrease in strength between -200 and +200°C, since no measurable change in elastic modulus occurs in this temperature interval. A "growth" of pre-existing flaws under the combined influence of stress and thermal fluctuations, as was previously suggested for germanium, appears to be a reasonable possibility.

A number of the features of the experimental work described above merit more detailed examination, in the light of the proposed explanation of the fracture behavior of these materials. The increase in the strength of 45° and 60° rods (Figure 7) above 600°C occurs at approximately the expected temperature, since macroscopic plastic deformation is observed above 900°C at stresses much smaller than would be expected near the tip of a potential crack. Two points of difference between these rods and the 0° rods (Figure 7) require explanation. First, the 0° rods appear to be about 50% stronger at liquid nitrogen temperatures. Since the tendency of sapphire to cleave is very slight, a large anisotropy in the fracture strength of bulk material would be rather surprising. On the other hand, Steijn's work on wear³¹ suggests that there may be a large difference in type and extent of surface damage for specimens of different orientation, and as a matter of fact, centerless grinding of 0° rods corresponds to the orientation and rubbing direction of minimum wear. Thus the high strength at low temperatures may simply reflect a difference in surface preparation, rather than a fundamental property of the material. The second point requiring examination, is the decrease in strength of these rods above 200°C, below that observed for skew-oriented rods. A tensile stress applied to a 0° rod has no resolved shear components on any of the known slip systems of sapphire. While it is local stresses caused by surface defects which are of primary concern here, it does not seem unreasonable to expect relaxation by plastic deformation to occur less readily in this case. Thus it seems likely that limited plastic relaxation occurs even at very low temperatures (at least in the skew-oriented rods), and that the fracture strength is determined by competition between two stress-dependent, thermally activated processes.

The results of Brenner²⁶ on whiskers (which would also have a 0° orientation) seem at first to contradict the results discussed above, since he found fracture stress decreased monotonically from room temperature to the melting point. Two important differences must be considered, however. First, these specimens would have had a nearly perfect surface, since a gauge section was produced prior to testing by thermal etching. Secondly, the orientation of whiskers undoubtedly would be more precise than would be possible with bulk specimens. Although the fracture stresses observed are much higher than those obtained in bulk specimens, they are still substantially below the theoretical

strength, and some defect undoubtedly acts to initiate fracture. The absence of the 600°C minimum observed in bulk specimens may reflect the ineffectiveness of basal slip in relieving stress concentrations caused by such a defect when the tensile axis coincides precisely with the c-axis of the specimen. An apparent inflection point in the fracture stress vs. temperature curve obtained by Brenner at 1550°C occurs close to the temperature at which slip on the prismatic system might occur. This system should be equally ineffective in relieving stress concentrations, however, and the origin of the inflection point is not clear at present.

The similarity of the results obtained for bulk specimens broken in tension²³ and in bending²⁴ (see Figure 7) suggests the relative unimportance of shear stresses in fracture nucleation, since the stresses quoted in each case were the maximum tensile stresses in the specimens at the time of fracture.

The difference in the behavior of single crystals and polycrystals (Figures 7 and 8) in the 600° to 800°C temperature range may reasonably be accounted for by the inhibition of plastic deformation by grain boundaries, which would otherwise act to relieve stress at a potential crack nucleus. This interpretation could readily be tested by determining the effect of grain size on the fracture strength curve in this range.

The experimental results relating to wear and friction discussed above bear out the hypothesis that plastic deformation, at least on the basal system, may occur at very low temperatures. It should be pointed out, however, that the etching results reported by Steijn,³¹ from which he concluded that plastic deformation due to rubbing penetrates to a depth of more than 25 microns at room temperature, could equally well be explained by a "dislocation crack" phenomenon, such as was described in a previous section for germanium. The amorphous nature of the wear debris is a rather compelling argument for plastic deformation, however. The stresses encountered in friction are likely to be predominantly of a compressive nature and may thus be quite large, but it should be recognized that the stress at the tip of a growing crack must represent the ultimate strength of the material, and that if plastic deformation occurs in the first case, it should probably be expected in the second.

The qualitative features of the frictional behavior of sapphire^{32,33} seem to be adequately accounted for by the assumption that both the minimum in the coefficient of friction which occurs at about 600°C in air, and the change from "slip-stick" to smooth friction which occurs at about 300°C in vacuum, relate to the onset of plastic deformation extensive enough to prevent brittle rupture of the bonded surfaces. The coefficient of friction in the first case could first decrease due to plastic deformation of the junction permitting easier rupture, then increase as plastic flow became more extensive, permitting larger junctions to be formed (independent of shearing). Some difference in temperature for the onset of deformation in the two cases (air and vacuum) would be expected because of the higher stresses which would result from the larger coefficient of friction in the latter case. Etching studies reported by the authors³² appear to support this interpretation. Evidence was reported for dislocation generation in 600°C specimens. Limitations of the etching technique are such that plastic flow confined to a thin junction layer could not be

detected, so deformation doubtless occurs at temperatures somewhat below 600°C.

Thus it appears that the fracture behavior and probably the dislocation kinetics of these two materials are very similar, at least up to about 60% of the melting temperature. The "model" proposed here, that of a thermally activated "growth" of pre-existing defects competing with plastic relaxation of the stress concentration to determine fracture strength, is a substantial departure from most of the current theories of fracture, which envisage dislocations acting in the opposite sense, to nucleate fracture. The nature of the growth process is a matter of speculation at present and is obviously an area in need of careful study.

It is suggested that at least one feature of the present treatment may have essentially universal applicability. It is energetically unfavorable for a dislocation whose stress field would be such as to further concentrate stresses at the tip of a crack to move into the stress field of the crack. In general, dislocations can be expected to intensify a stress concentration only when forced into such a region by other dislocations. Stroh³⁴ has discounted the effectiveness of stress relaxation (the energetically favorable process) by plastic flow at the crack tip on the grounds that spontaneous dislocation nucleation in bulk material requires stresses near the theoretical strength of the material and hence should not be expected to occur. However, generation of dislocations at a crack tip certainly must require smaller stresses than are needed to rupture the bonds there and cause the crack to propagate. Furthermore, Hirth³⁵ has recently shown that surface structure of a type which reasonably might be expected to occur in a microcrack can result in a reduction of the stress required for spontaneous dislocation nucleation by a factor of 2 or 3 for copper and aluminum, and presumably for other crystals as well. Thus it would seem that plastic relaxation should always occur before fracture nucleation by dislocation pile-ups. As previously pointed out, the effectiveness of the relaxation process will be limited by the local nature of the stresses at the crack tip, so that fracture strength will again be determined by competition between two or more kinetic processes, resulted in the observed complex temperature dependence of the fracture strength.

Part C: THE EFFECT OF ATMOSPHERES ON CREEP

I. Introduction:

Many of the models which have been proposed to explain creep in single crystal and polycrystal material should be influenced by non-stoichiometry. One can see several reasons why either the transient or the "steady state" creep should be influenced by vacancies and/or interstitials.

Creep was measured in Al_2O_3 single crystal and polycrystal samples that had been given different atmospheric heat treatments. The procedure was to see if there was any consistent variation in a number of samples with the same thermal history in different atmospheres.

II. Experimental Procedure:

The single crystal bend specimens were made from 40 mil diameter Linde single crystal 60 degree oriented rods, 25 cm long or longer. The rods were selected for their perfection, oriented so that the C-axis was in the plane of bending, and ground flat on the ends by a 160 mesh diamond wheel so that the rods would remain in the same orientation during bending.

The comparison of the atmosphere effect on single crystals was accomplished by numbering the specimens taken from a 10 inch rods that had flat spots ground every inch while the rod was still intact.

The odd numbered specimens were then heat treated in an oxygen atmosphere while the even numbered specimens were treated in nitrogen. In this manner any systematic orientation changes down the length of the rod could be detected.

The polycrystalline specimens were prepared from sintered discs having a grain size of 8-10 microns with greater than 95% theoretical density. They were very accurately ground to $.75 \times 1.5 \times 20 \text{ mm} \pm .003 \text{ mm}$ with diamond cutting and grinding wheels. The sintered discs had been prepared by Lew Cook of the ceramic engineering department and had impurities of less than 100 ppm.

The prepared samples were then heat treated at temperatures from 1350-1580°C in a furnace where an atmosphere of O_2 , N_2 , or Argon with a dew point of less -70°C could be maintained.

The creep rates were measured in a four point loaded furnace of the type previously described by Beauchamp, et al³⁶ but with modification permitting atmosphere control. The outer knife edges were 1.6 cm apart and the inner knife edges were .552 cm apart. The knife edges were accurately positioned symmetrically by a specially made aluminum "key."

The strain was detected by a linear variable differential transformer (Schaevitz) operated at 500 cps. The output of the LVDT was rectified and recorded on a Brown recorder. The deflection sensitivity was .66 microns which amounted to a sensitivity of 10^{-5} outer fiber strain on the samples used.

The specimens were loaded hydraulically by allowing the distilled water surrounding a symmetrical float to vary in height. The geometry of the float and the size of the surrounding cylindrical plastic tank was such as to allow a very rapid application of load with almost no overshoot.

Single crystal specimens were heat-treated at temperatures of 1350°C, 1450°C, 1580°C for times of up to 50 hours. The specimens were then removed from the heat-treating furnace with a rapid air cool. The specimens were deformed in the creep furnace at 1350°C in the same atmosphere within 40 minutes.

III. Results:

Creep curves for the single crystal specimens treated at 1580°C are shown in Figure 9a. The curve labeled I is for the number one and two specimens taken from a rod and heat-treated 24 hours in $O_2(o)$ and $N_2(x)$ at 1580°C. The curve labeled II is for the number three and four samples from the rod which were treated 50 hours at 1580°C. The curves labeled III are the number one and two samples taken from another rod with a 50 hour 1580°C atmosphere heat treatment. The maximum outer fiber stress was 1×10^4 gm/mm² in all cases. The delay times are almost identical and the strain rate varies by less than 8% at the worst portion of the curve, and is almost identical at the low and high values of strain.

Twelve single crystal specimens were selected for the 1450°C - 24 hour heat treatment. Five were in an O_2 atmosphere, five in N_2 and two in argon. The results are shown in Figure 9b where the maximum and minimum curves are shown for 12 samples. The loading was greater (1.13×10^4 gm/mm²) to give a greater initial strain rate and the results were almost identical, particularly at low and high values of strain. There was less than 6% variation and the averages of the three groups are almost identical.

The 1350°C - 24 hour heat treatment results are shown in Figure 9c. Stress was 1.13×10^4 gm/mm². There is more scatter in these measurements with only three N_2 and three O_2 samples. However, analysing the average of the initial slopes it was found they varied by 6.5% and the average of the final slopes varied by 2%. The standard deviation of the oxygen initial slope amounted to 10% and the standard deviation of the final slope amounted to 4%. Thus even for this group the average values of the three slopes were within the standard deviation.

The results for six polycrystalline samples given 1450°C - 24 hour treatments in O_2 and N_2 are shown in Figure 10a where the maximum and minimum curves are shown. Stress was 7.5×10^3 gm/mm². They were loaded and unloaded twice during the creep run to see in particular if there was any change in the transient. There was no variation in the initial slopes and the variation in the first steady state creep was 10% with no consistent atmosphere effect. The later transients and steady state creep rates were almost identical.

The results for six polycrystalline samples which were heat treated for 50 hours at 1580°C are given in Figure 10b. Stress was 7.5×10^3 gm/mm². Again there was no consistent variation in strain rates.

There are variations between samples with the same atmosphere heat treatment that are greater than the variations in the average of the two groups of samples. A larger number of samples would be needed to determine the statistical correlation. The influence of atmosphere was never greater than the variation in the samples which was as low as 6%.

IV. Discussion

Experimentally it has been shown that there is no large influence on the strain rate by variations in atmosphere.

These negative results are consistent with the lack of influence that foreign atoms have on creep found by Beachamp et al.³⁶ or on sintering rates as shown by Cutler et al.^{37,38} More directly, the presence of hydrogen or oxygen atmospheres cause no change on the sintering rates, even at 1500°C, as shown by Coble³⁹ and Cutler, Jones, Maitra.⁴⁰

The electrical conductivity experiments at high temperatures as a function of oxygen partial pressure by Pappis and Kingery⁴¹ indicate (however not conclusively) that Al^{+++} vacancies are found in high O_2 pressures and Al^{+++} interstitials or O^{--} vacancies at low oxygen pressures. Thus Al_2O_3 can become non-stoichiometric but the point defects are probably associated with the smallest ion.

All of these results seem to be consistent with the idea that either the type of defect produced or the number of defects produced is insufficient to produce any significant change in the diffusion of oxygen or a change in the dislocation creation or motion. These results would be consistent with the possibility that Al^{+++} interstitials or Al^{+++} vacancies are the point defects that are formed for non-stoichiometry. As Kingery has pointed out, the crystal structure in which two-thirds of the available octahedral sites are filled by Al^{+++} and the structure of $\gamma-Al_2O_3$ in which a randomly distributed fraction of the cation sites remains unfilled are in agreement with this possibility.

One can argue qualitatively that Al^{+++} interstitials would certainly decrease the oxygen diffusion and the presence of oxygen vacancies would increase the oxygen diffusion. These two alternatives in light of the experimental data point to Al^{+++} interstitials as the point defect.

Since the size of the oxygen ion is 2.4 times the size of the aluminum ion, one would also most likely consider that the oxygen vacancy would have a greater pinning effect than the aluminum interstitial. Thus the negative effect on the transient and steady state creep points also to Al^{+++} interstitials rather than O^{--} vacancies.

As an upper bound one might expect less than $10^{17} - 10^{18}$ point defects to be produced by different atmospheres at these temperatures. If these are randomly distributed, they ought not to produce a greater effect than that due to impurity studied by Beauchamp et al.³⁶ A much greater effect would be expected if they were formed in the neighborhood of and served to pin potentially active dislocation generators.

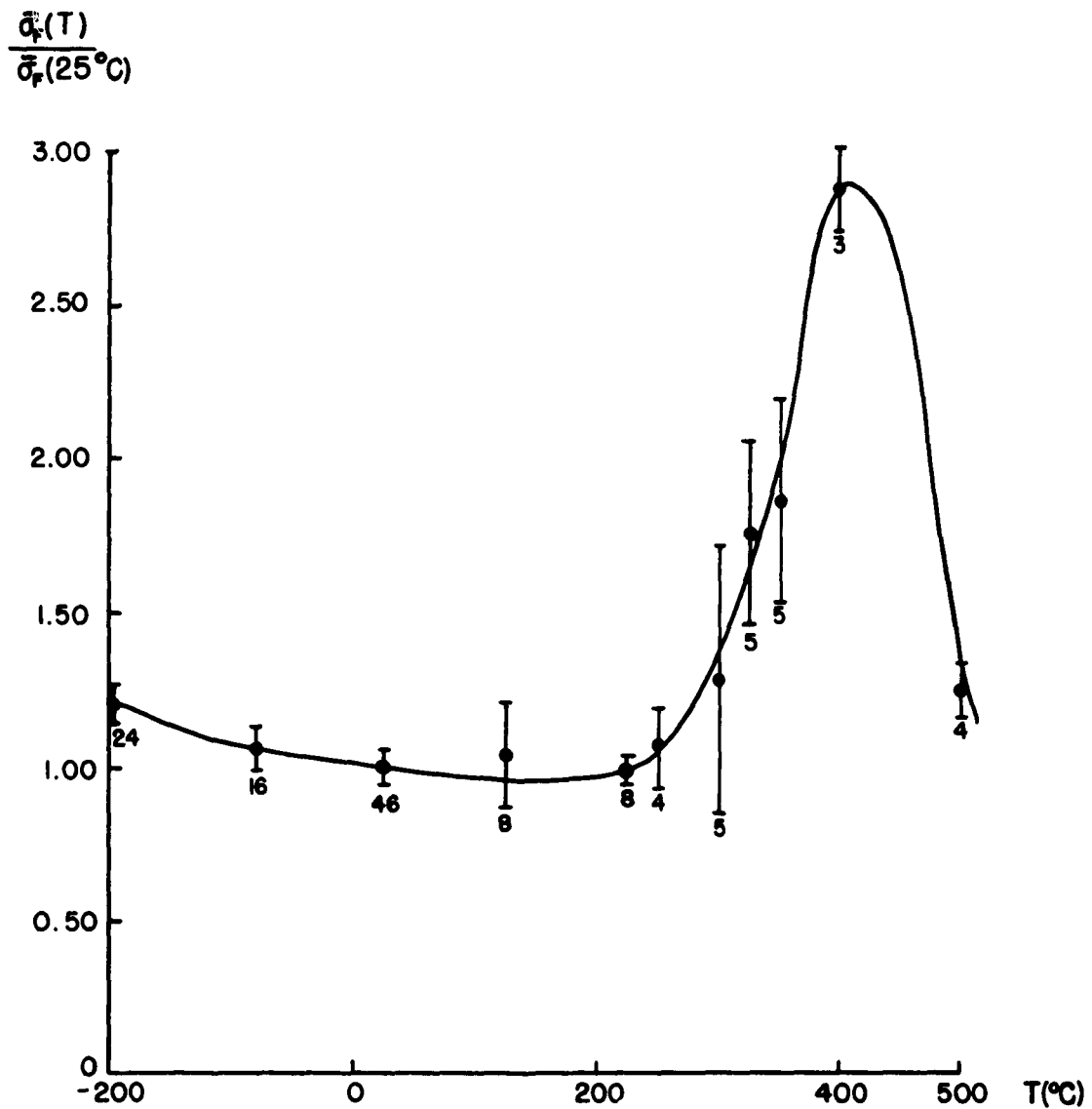


Figure 1

Variation of average fracture stress ("normalized") of germanium with temperature. Flags indicate 5% uncertainty limits. Numbers below data points indicate number of specimens tested.



Figure 2
Fracture nucleus, lightly etched, showing initial stage of dislocation generation. Specimen broken at 300°C. 1460X.



Figure 3a

Fracture nucleus, lightly etched, showing moderate plastic deformation prior to fracture. Specimen broken at 400°C. Rounded arrays of pits (upper and lower right) are shallow arrays of imperfections produced during fracture. 400X.



Figure 3b

Same crystal as Figure 3a after removal of about 6 microns by polish-etching, then re-etching in dislocation etchant, showing limited depth of the rounded arrays of imperfections shown in Figure 3a. 400X.



Figure 4

Fracture nucleus, lightly etched, showing extensive plastic deformation prior to fracture. Specimen broken at 500°C. 400X.

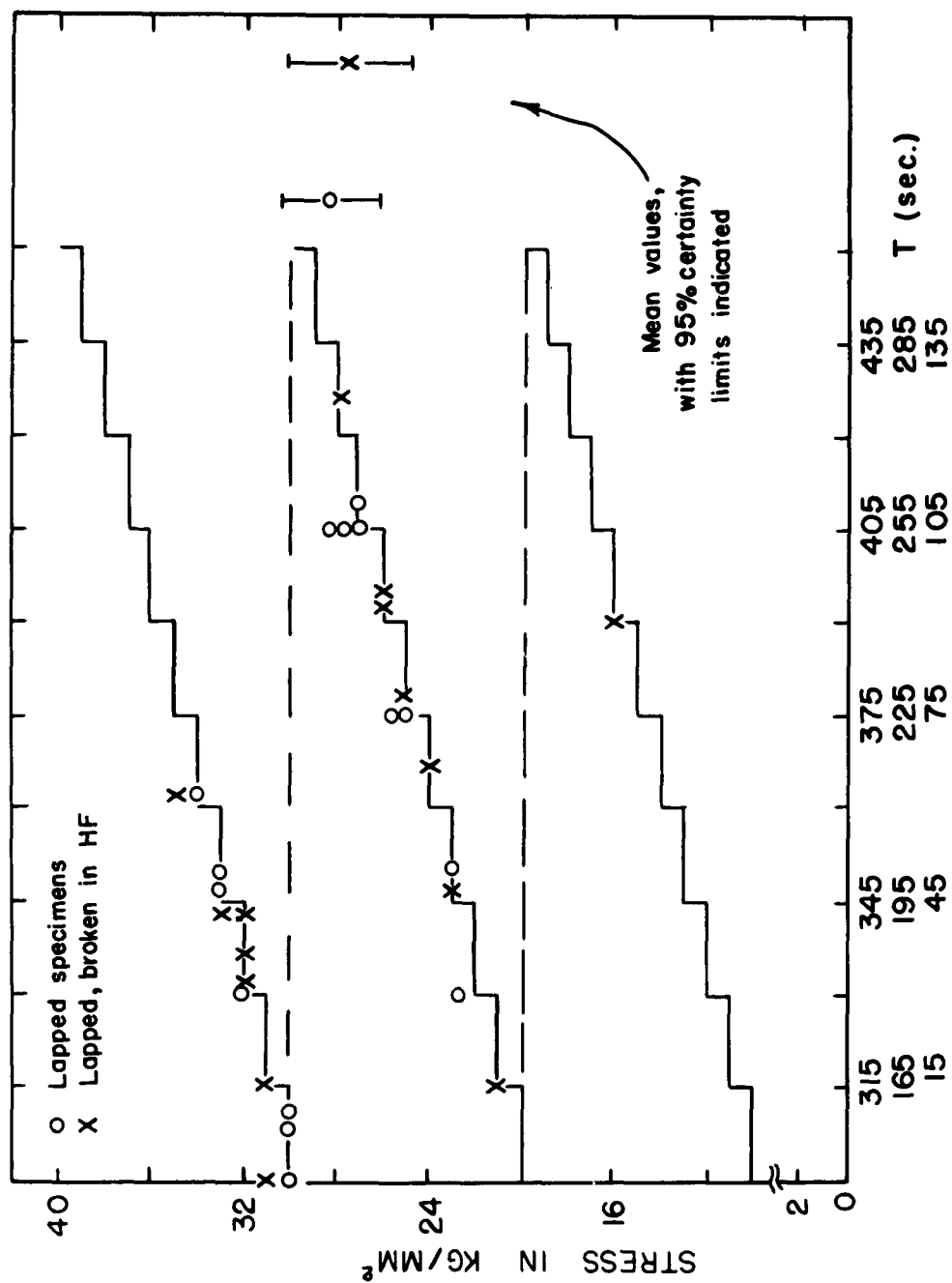


Figure 5

Fracture stress and delay times for specimens broken in HF acid in air. Solid line is "loading curve," indicating applied stress as a function of time. Mean values indicated at right.

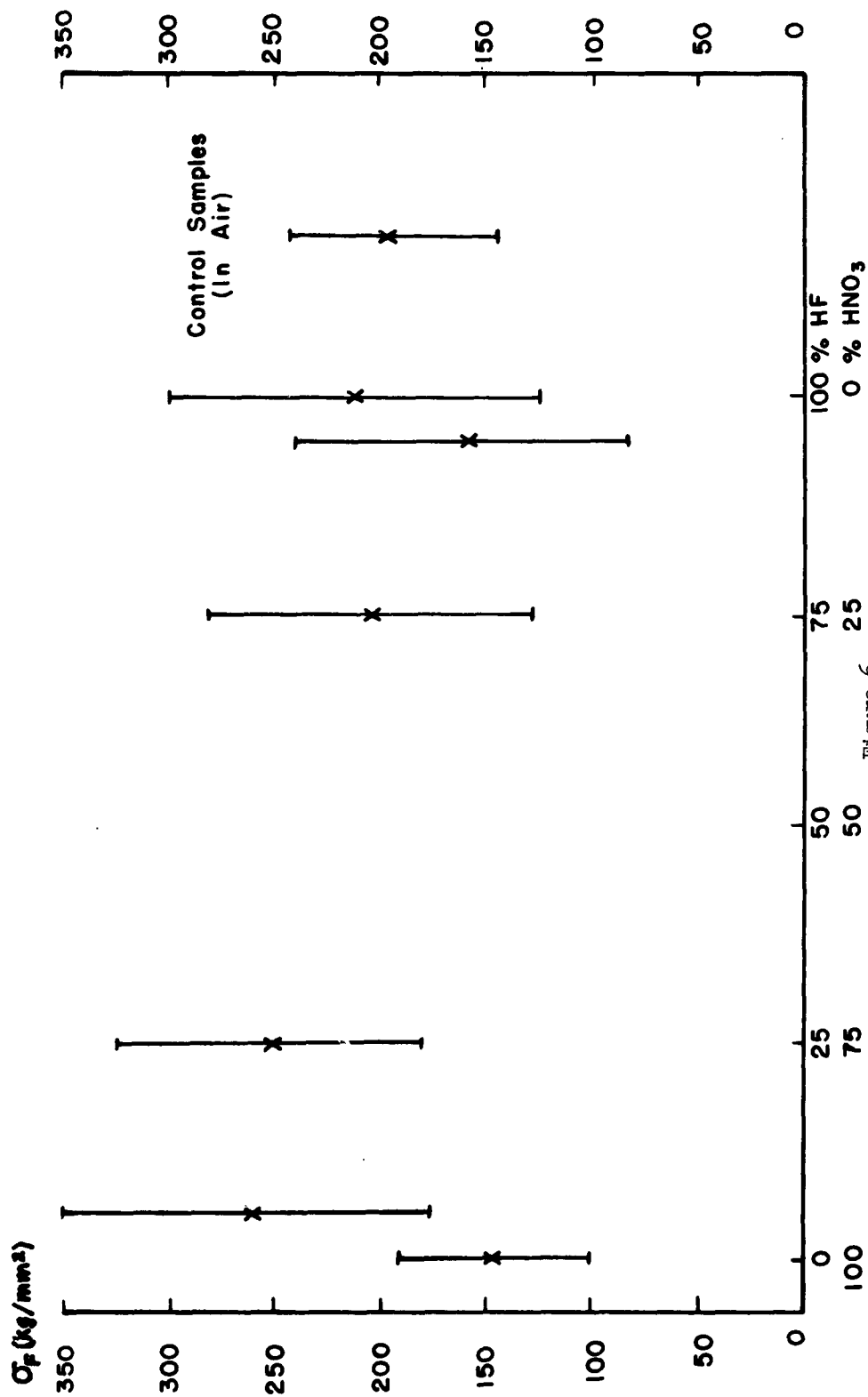


Figure 6
Average fracture strength for various proportions of HF and HNO_3 . Control samples at right. 5% uncertainty limits indicated.

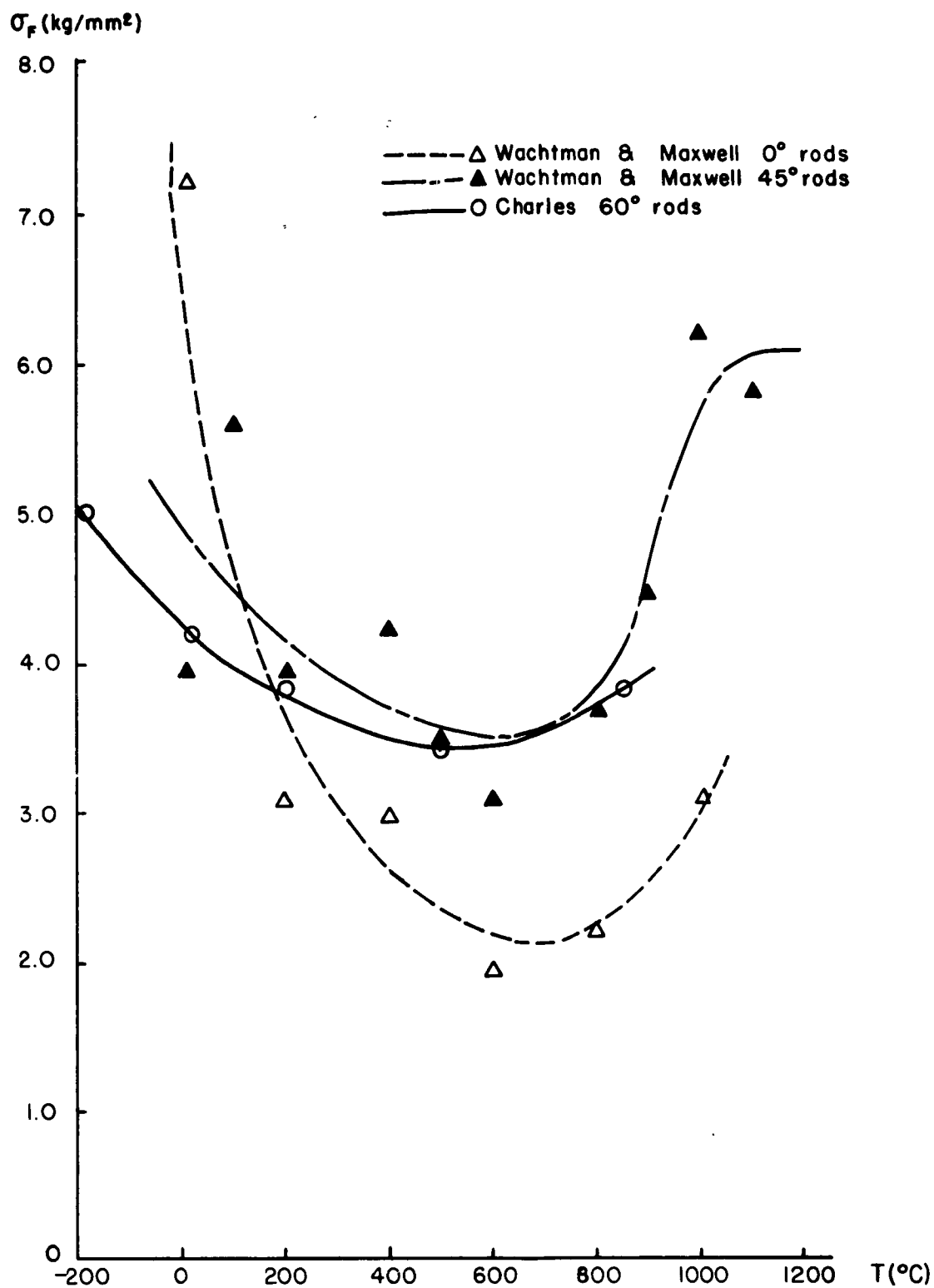


Figure 7
Variation of average fracture strength of sapphire with temperature
(after Wachtman and Maxwell²³ and Charles²⁴).

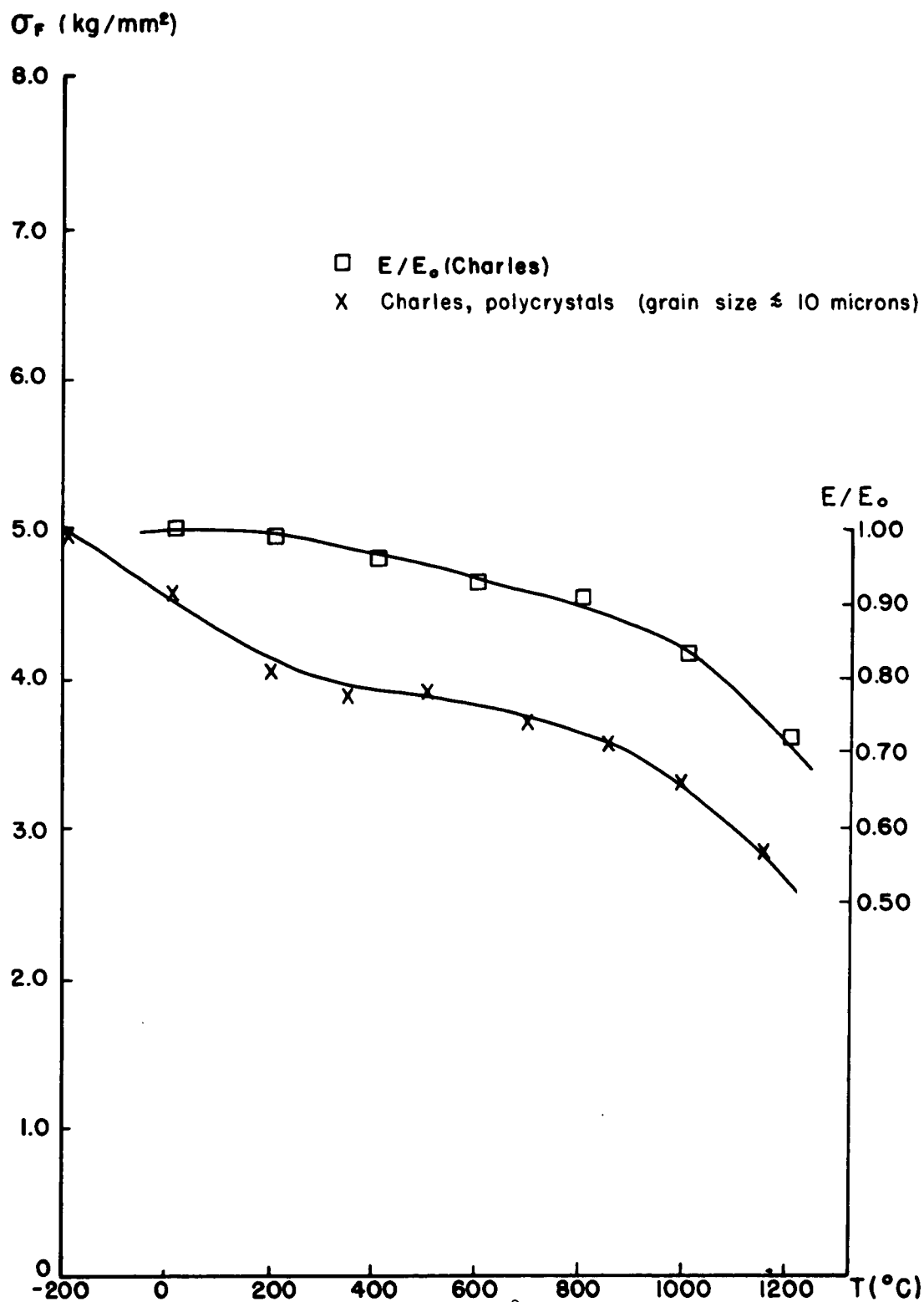
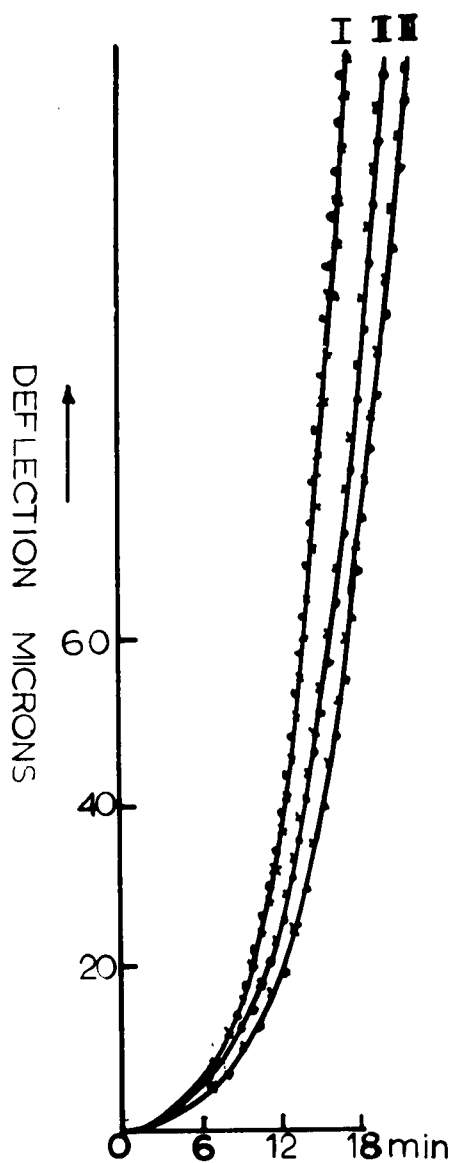
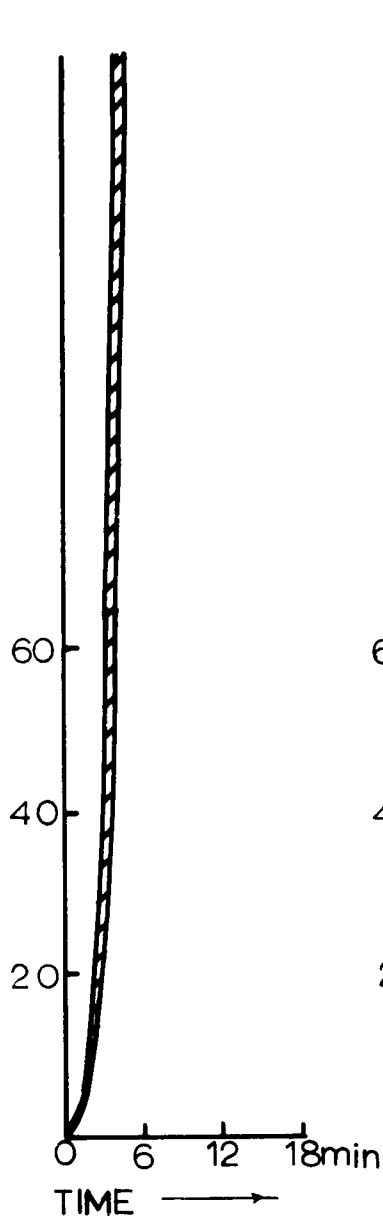


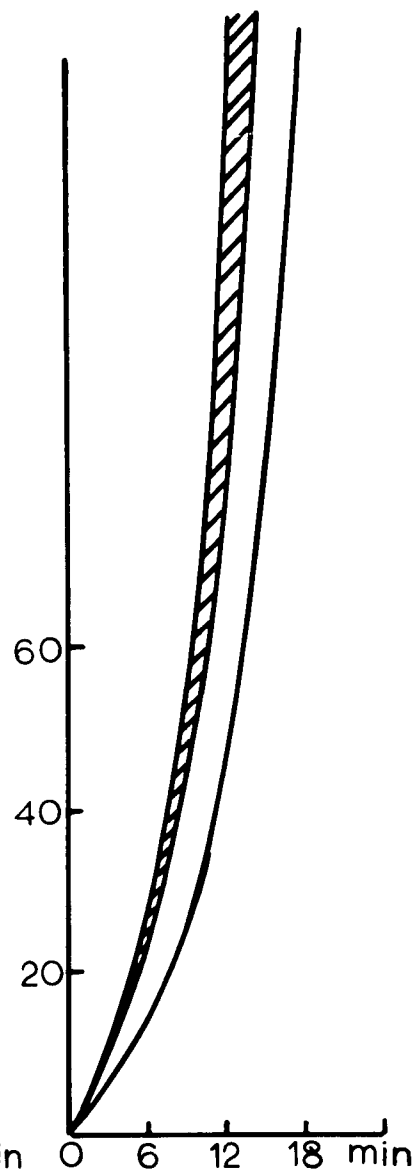
Figure 8
 Variation of average fracture strength and elastic modulus of polycrystalline Al_2O_3 with temperature (after Charles²⁴).



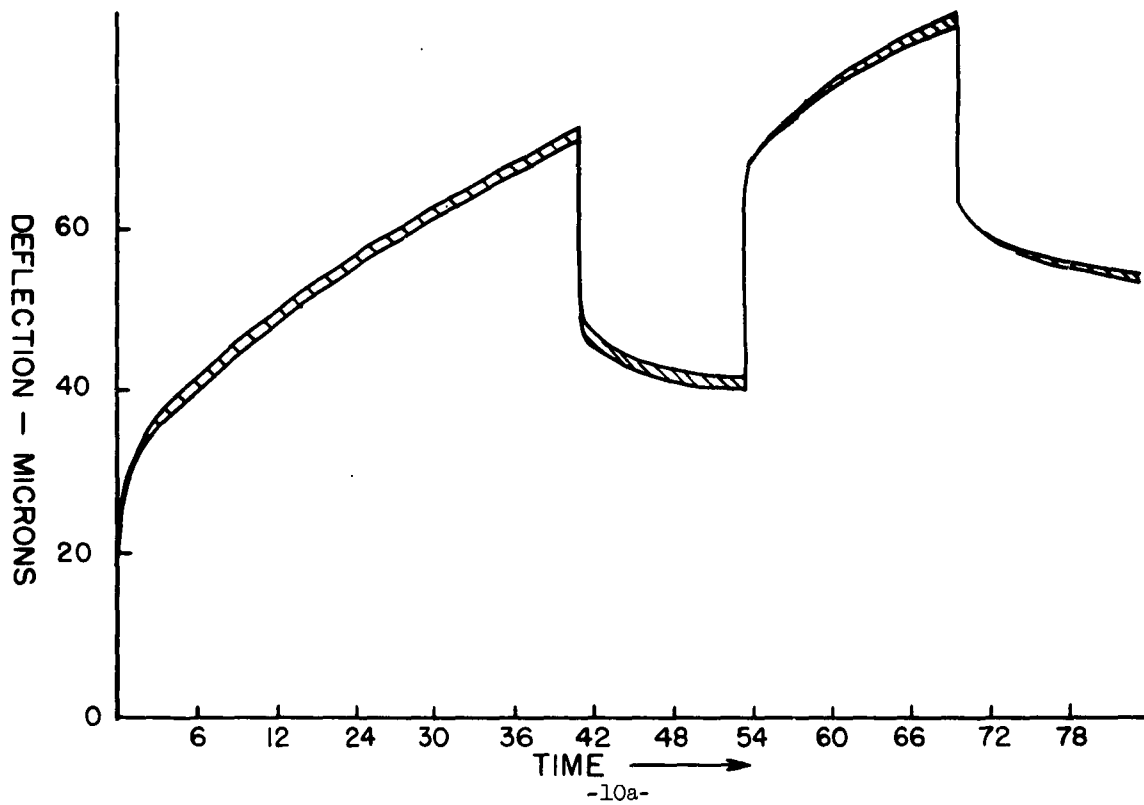
9a
Specimens treated in
 O_2 or N_2 at $1580^\circ C$.
See text.



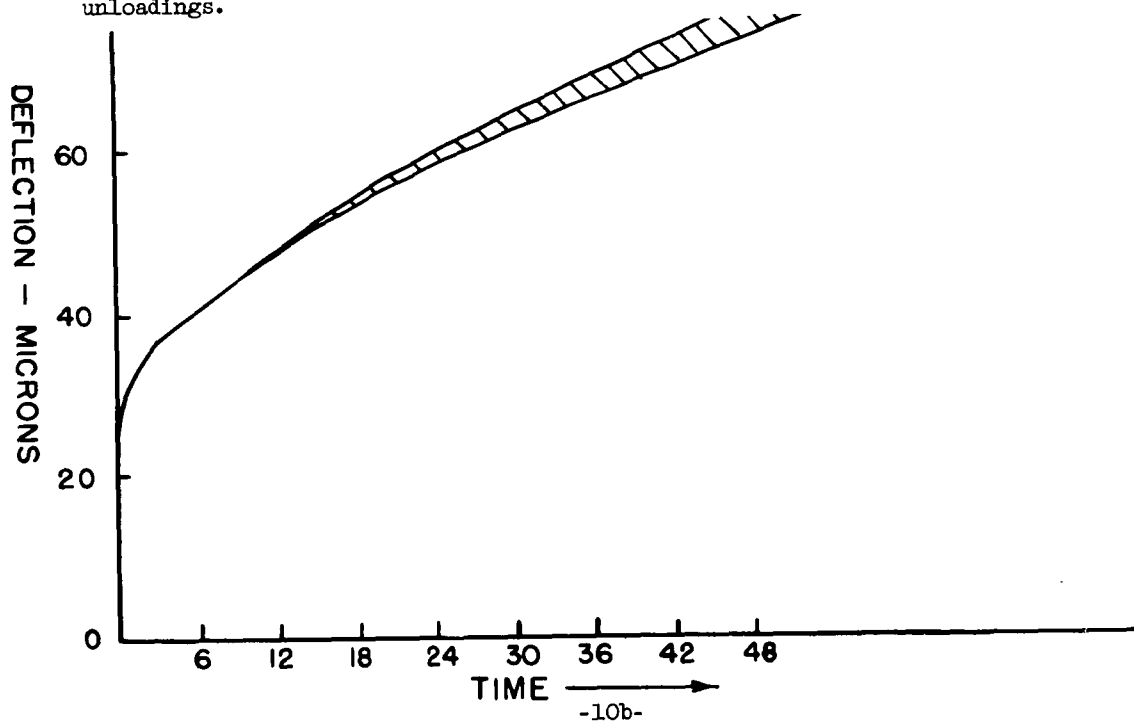
9b
Maximum and minimum creep
curves for 12 samples hea-
ted in O_2 , N_2 and argon at
 $1450^\circ C$ for 24 hours.



9c
Maximum and minimum creep
curves for 6 samples given
24 hours, $1350^\circ C$, O_2 and
 N_2 heat treatment.



Maximum and minimum creep curves for six polycrystalline samples heat-treated for 24 hours at 1450°C in O_2 and N_2 showing the results for two loadings and unloadings.



Maximum and minimum creep curves for six polycrystalline samples given 50 hours, 1580°C, O_2 and N_2 treatment.

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Unclassified Report

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(over)

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4. Creep
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II. Contract AF 33(616)-
6892

III. University of Utah,
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I. AFSC Project 7350,
Task 735001
II. Contract AF 33(616)-
6892
III. University of Utah,
Salt Lake City, Utah
IV. O. W. Johnson, M.
Farb. P. Gibbs
V. Avail fr OTS
VI. In ASTIA collection