DEFORMATION PROCESSES IN FORGING CERAMICS

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ABSTRACT

The program objective is to investigate the deformation processes involved in the forging of refractory ceramic oxides. A combination of mechanical testing and forgings is being utilized to investigate both the flow and fracture processes involved.

Initial forging experiments were conducted during this quarter. The microstructural examination of these and comparison with flexural specimens tested at 1450°C indicates deformation proceeds by grain boundary sliding and that slip is important in the grain shape change necessary for conformity. A strong crystallographic texture was found in a fine-grained specimen forged at 1450°C.

Investigation of the fracture stress of fine-grained alumina revealed a strong dependence of the fracture stress on strain rate in the strain rate range where there is little deformation preceding fracture.

FOREWORD

This work is being performed under the sponsorship of the NASA Headquarters, Office of Advanced Research and Technology, Research Division with Mr. J. Gangler as Project Monitor under Contract NASW-1914.

The work is being performed at the Avco Corporation, Systems Division, Lowell, Massachusetts in the Materials Sciences Department under the direction of Dr. T. Vasilos. Mr. R.M. Cannon is in charge of the work and is being assisted by Dr. W.H. Rhodes. The authors wish to acknowledge the assistance of Mr. B. MacAllister in Mechanical Testing and the microscopy of Mr. P.L. Burnett and C.L. Houck, and R.E. Gardner and of P.L Berneburg and R.M. Haag in X-ray Diffraction.

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I. INTRODUCTION

The objective of this program is to investigate the forgability of the refractory oxides. The approach taken includes an investigation of the high temperature yielding and fracture behavior of these materials in order to provide information and understanding which can then be applied directly to forging problems. The primary emphasis has been on mechanical properties studies. A few forgings are being done to supplement the mechanical testing results.

The first progress report, included reviews of the work and understanding to date of the high temperature mechanical behavior of the oxides as well as hot working efforts with these materials.[1] On the basis of this study, two systems were identified for primary investigation. These were fine-grained alumina, doped to inhibit grain growth, and magnesia, which will probably require temperatures in excess of 2100°C in order to obtain sufficient tensile ductility. Spinel ($MgAl_2O_4$) was tentatively selected for later investigation.

The preliminary results of a series of mechanical tests on alumina done by reversed, multiple loadings in flexure were reported in the last two reports.[1,2] Multiple loading in flexure was done in order to investigate the effects of multiple step deformation, intermediate annealing and reversed loading on the mechanical behavior. These various effects are particularly relevant to forging and also provide insight into fundamental mechanical processes and mechanisms. The investigation has included extensive microstructural as well as mechanical observations. The results have been interpreted in terms of a combination of grain boundary sliding and slip.

During the present reporting period, the flexure testing has been continued and includes some investigation of fracture as well as deformation. In addition, several forgings of alumina have been done in order to broaden the mechanical behavior studies and to indicate some of the engineering problems associated with forging. Two upset forgings and one deep drawing of a hemisphere were done with alumina. Microstructural examination of these forgings has provided further insight into both fracture and flow mechanisms; they have generally supported the results of the flexure tests.

II. MECHANICAL TEST RESULTS

During this reporting period the testing has been completed for the multiple bending series which was described in the last report.[2] The final analysis of the data to correct for curvature and other effects associated with the bend test have not yet been completed. In addition, testing has been initiated on a series of tests designed to clarify the dependence of strain rate and stress on grain size. Finally, a group of tests to indicate the effect of strain rate on fracture stress is in progress. All of these are being done with alumina with additives to inhibit grain growth.

Testing is done in four-point flexure in an argon atmosphere furnace. The load is recorded continuously versus time. Deflection is measured with a probe system monitored with an LVDT which is also continuously recorded versus time in order to be able to determine stress, strain and strain rate continuously throughout the test.

A series of tests to provide insight into the fracture mechanisms in alumina are being conducted. A series of constant strain rate tests at 1450°C have been run over a range of strain rates; the range is sufficiently broad that essentially brittle fracture results at the high end and fully plastic behavior is obtained at the low end. Although testing is not yet complete, the results to date are presented in Figure 1. These specimens were all taken from a single billet, C79, of Al_2O_3 + 0.1% MgO hot pressed to over 99.5% density. The points marked fracture stress are for specimens which failed with almost no plastic strain since the steady-state flow stresses would have been considerably in excess of the fracture stress. The points marked yield stress are for specimens which did not fail when bending was terminated after 4% strain since the flow stress was below the fracture stress. These have subsequently been rebent several times without failure. These data indicate that there is a strong dependence of fracture stress on strain rate even in the range where the total strain to failure is small.

Final interpretation of these results is not warranted until all of the tests are completed and microstructural examination is done. It is suggested that at the high strain rate the fracture occurs in an essentially brittle manner similar to that which occurs at lower temperatures. As the strain rate is lowered, the flow stress is reduced so that small amounts of strain can occur which result in a reduction of the fracture stress. Relaxation of grain boundaries resulting in increased stress concentrations is thought to be one possible explanation for this. The possibility of limited slip providing a method of crack nucleation is also being considered. Additional tests are in progress to complete the curve in Figure 1, and microstructural and fractographic examination will be conducted. The final analysis will attempt to differentiate between the effects of strain rate per se and the amount of strain at failure which is influenced by the strain rate.

III. FORGING

Three forgings of fine-grained alumina were done. Two of them were simple upset forgings which were designed primarily to provide an assessment of microstructural effects to complement the mechanical tests. The third was an effort to deep draw a 2-inch diameter hemisphere from a 3-inch blank. All of these forgings also provided valuable assessment of some of the engineering problems associated with forging which will be useful in attempts to forge more complicated shapes. All three forgings were done on doped Al_2O_3 of 1 to 2 μ grain size which had been hot pressed to greater than 99.5% density.

The two upset forgings were done in a modified hot pressing apparatus arranged so there was no side constraint on the pieces. The press is hydraulically driven so that precise speed control is not possible. However, the deflection is monitored by a dial gage, and load was continually adjusted to provide a nominal strain rate of 4×10^{-5} sec^{-1} for both tests. They were both done at 1450°C. These conditions are similar to those in

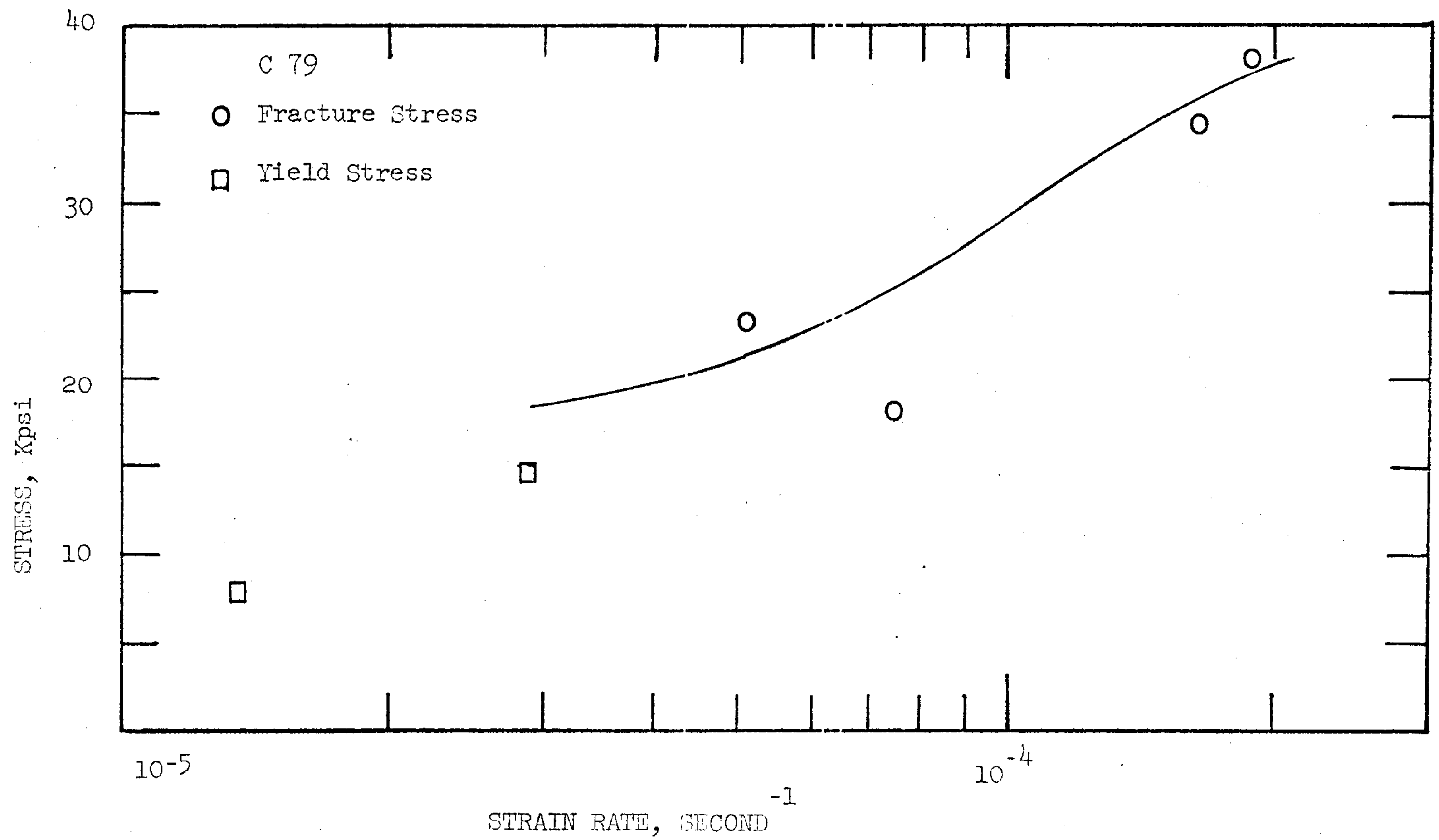


Figure 1. Effect of Strain Rate upon Fracture Stress of Alumina at $1450^{\circ}C$

in which the bend testing has indicated highly rate-sensitive deformation resulting from grain boundary sliding with an apparent contribution from slip. This temperature is also low enough that rapid grain growth is not expected.

One of the pieces was forged to 16% height reduction and had a nominal L/D ratio of 0.72 at the end. This required a pressure of about 5,500 psi by the end of the stroke. This piece had no visible damage after forging. The second piece was forged to a reduction of 38% and required a pressure of about 10,700 psi by the end; it had a final L/D ratio of 0.63. The pressure difference between the two cannot be taken as direct evidence of hardening because of a difference in grain size of the two pieces and because of differences in die facing materials resulting in greater frictional effects in the 38% forging.

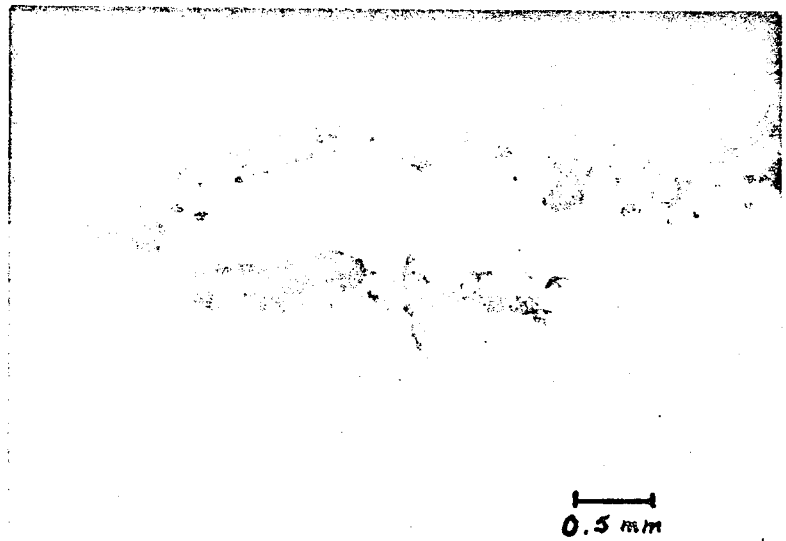
The 38% forging was somewhat asymmetric because of corner hang-up on one side. In addition, close examination of the surface revealed several small cracks on the sides, examples of which are shown in Figure 2. These cracks are associated with small regions containing several large grains. The large grains were extruded out from the surface as can be seen in the photographs. It is rather encouraging that the cracks did not propagate far from the defect areas.

An initial attempt to deep draw a 2-inch diameter hemisphere from a 3-inch disc was made. This was done at 1500°C with a target nominal strain rate of 4×10^{-5} sec^{-1}. This was also done in a modified hydraulic hot press. The load was increased in an attempt to maintain the desired travel rate. Several problems in maintaining the necessary control of the travel rate and load were indicated which will receive attention before the next attempt. The piece was broken in several pieces; preliminary analysis suggests that the cause was probably excessively high stresses resulting from failure to obtain proper travel and load control.

IV. MICROSTRUCTURAL EVALUATION

The two upset forgings have received rather extensive microstructural evaluation in order to further assess the flow and fracture mechanisms and to correlate these results with those from the mechanical testing. In addition, examination of the flexural specimens has continued with attention to both flow and fracture mechanisms.

Examination of the forged pieces revealed features similar to those previously reported[2] in the flexure specimens. The piece forged 16% revealed similar evidence of grain boundary sliding. An example is shown in Figure 3 of a chemically polished and etched cross-section. Evidence of offset or distorted triple junctions can be seen which are strong evidence for grain boundary sliding. In addition, it can be seen that the boundaries are frequently jogged; this is thought to be the result of dislocation grain boundary interactions. The 'double' boundaries (arrows) found in the flexure specimens are also in evidence here. These are thought to be an indication of the localized shear resulting from the boundary sliding; the



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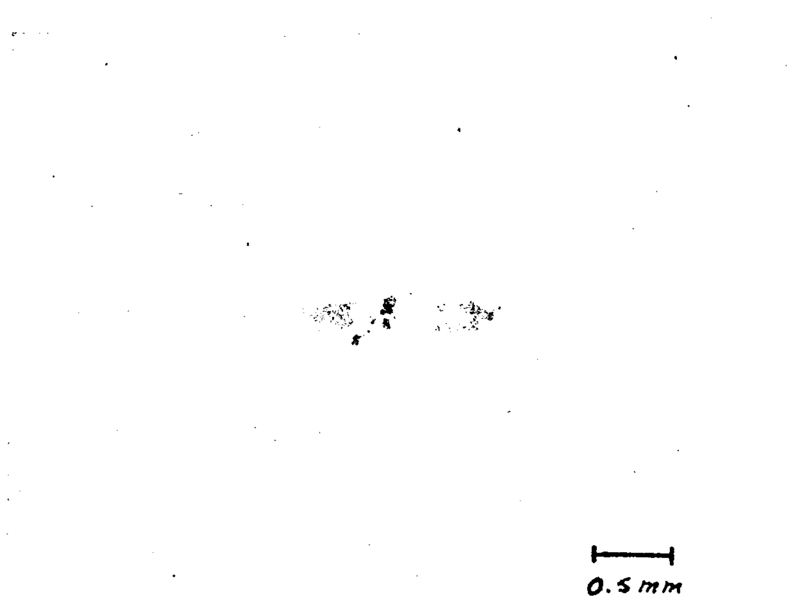

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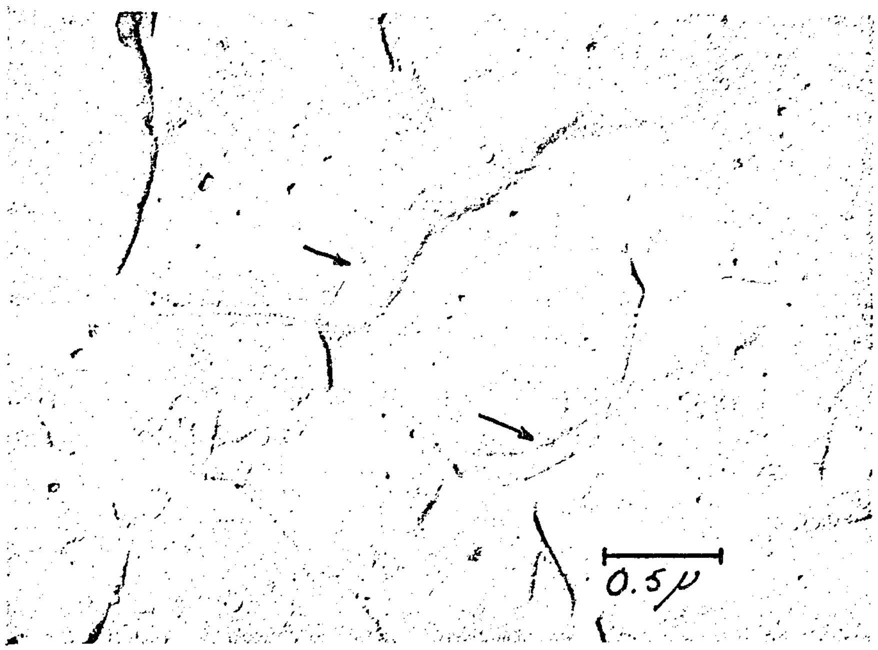
Figure 2. Small Cracks seen in the Side of Specimen JC-1474 After Forging to 38% Height Reduction. The Cracks are Associated with Small Patches of Coarse-Grained Material Which are seen to have been Extruded out of the Surface.



#70401 30,000X

Figure 3. Cross-Section of Sample JC-1469 Forged 16% at 1450°C. The Sample was Chemically Polished ($Na_2B_4O_7$) and Etched ($K_2S_2O_7$). Plane of the cross-section is parallel to the forging direction.

second boundary may be a low angle boundary or dislocation network. Some of the faint lines originating at boundary jogs or near triple points are suggestive of dislocation sources resulting from grain boundary sliding which have recently been observed in metals.[3-5] The previously reported observation, by transmission electron microscope of grain boundary dislocations in alumina after small strains, lends support to the suggestion of dislocation emission by sliding boundaries.

The piece forged to 38% reduction was in many regards similar in that offset triple points (arrow) and boundary jogs were present as seen in Figure 4(a). In addition, this specimen showed some microstructural texture from elongated grains which can be seen in Figure 4(b). This specimen also exhibited a considerable amount of intergranular porosity as can be seen in the micrograph.

In order to assess the contribution of slip to the deformation, the forged specimens were analyzed for preferred crystallographic orientation by an x-ray diffraction technique which has been developed on previous programs.[7] The crystallographic texture can be described by what is essentially an azimuthally averaged inverse pole figure. In this procedure the relative population density of different planes is plotted against the angle between these planes and the basal (000.1) plane. With proper normalization this is then also a plot of the population density of the basal planes at the same angle from the reference surface.

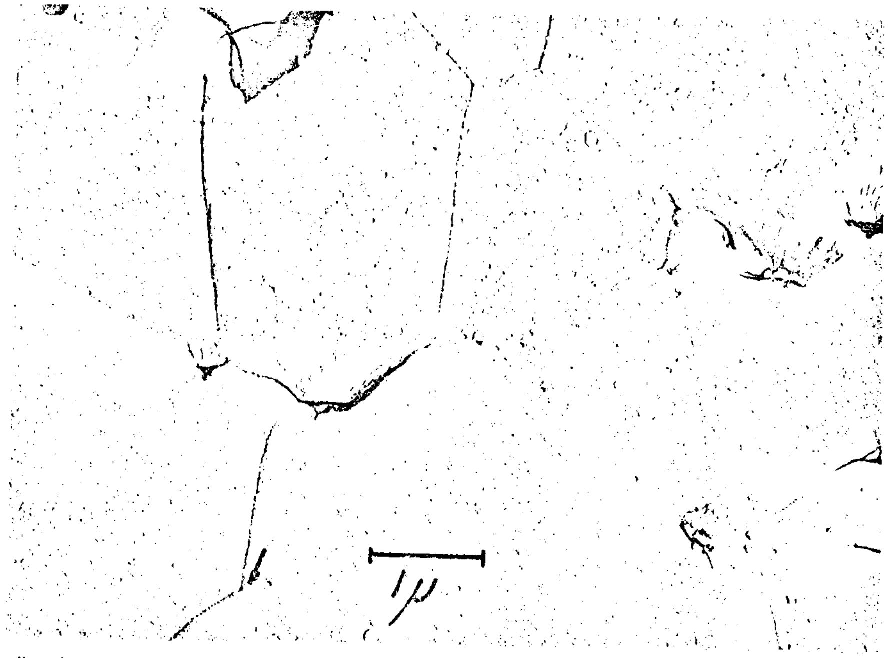
Experimentally, the procedure is simple. The diffraction pattern is a randomly oriented (powder) sample is obtained. Values are calculated of $P_o(hk \cdot l)$ defined by the relation

$$P_o(hk \cdot l) = \frac{I\ (hk \cdot l)}{\sum_{hkl} I\ (hk \cdot l)}$$

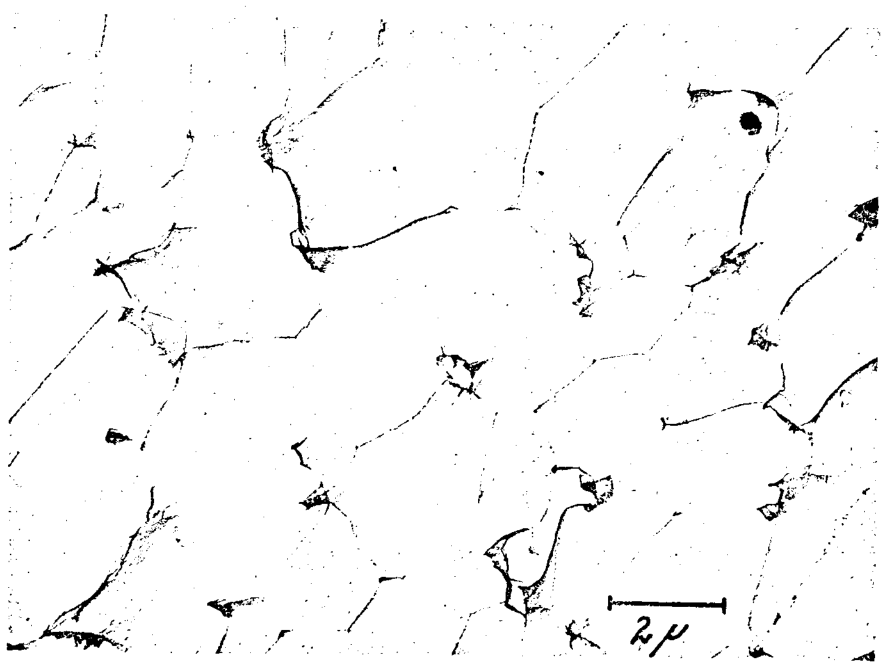
where $I\ (hk \cdot l)$ is the diffraction peak intensity for reflection from the $(hk \cdot l)$ planes. Similarly, the diffraction pattern of the forged body is obtained using a face perpendicular to the pressing direction. Values of $P(hk \cdot l)$ are calculated as before, and then the ratios $R(hk \cdot l) = P(hk \cdot l)/P_o(hk \cdot l)$ are calculated. These values of R are plotted against φ, the angle between the plane $hk \cdot l$ and 00.1.

In the case of a random (powder) sample, R has the constant value of unity. In the case of a perfectly oriented sample, R is zero everywhere except at $\varphi = 0$ where it has some large finite value. In the case of a distribution of orientation, R will, in general, decrease monotonically from $\varphi = 0$ to $\varphi = 90^o$. The better the alignment of the crystallites, the greater will be the intercept at $\varphi = 0$ and the steeper the drop with increasing φ.

The results for the 16% reduction sample are not yet available; however, the specimen forged 38% has a marked basal texture as shown in Figure 5.



#70408 (a) 15,000X



#70407 (b) 7,500X

Figure 4. Cross-Section of Specimen JC-1474 Forged to 38% Reduction at 1450°C. Specimen mechanically polished and etched (H_3PO_4). Cross-section is parallel to the forging direction.

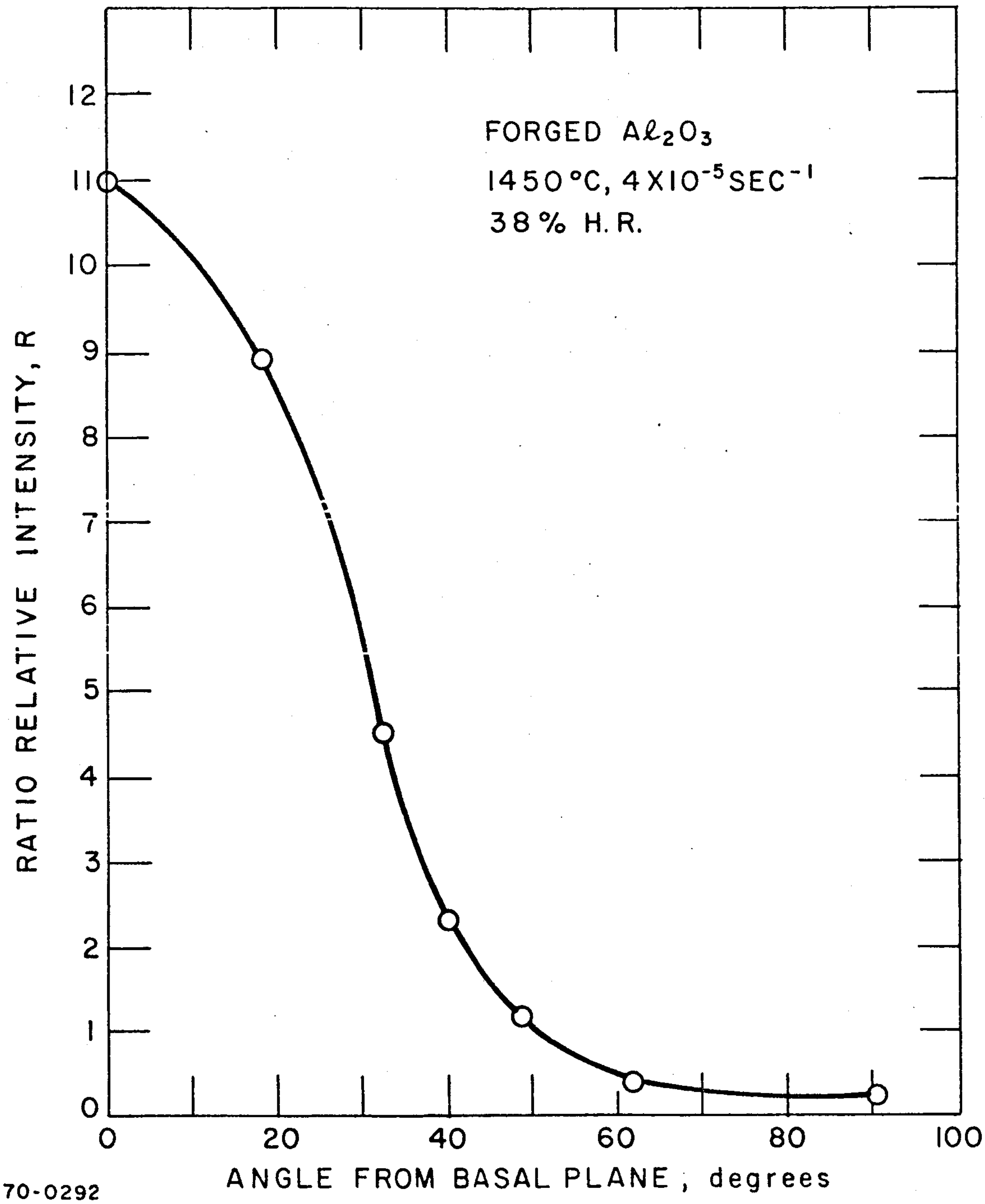


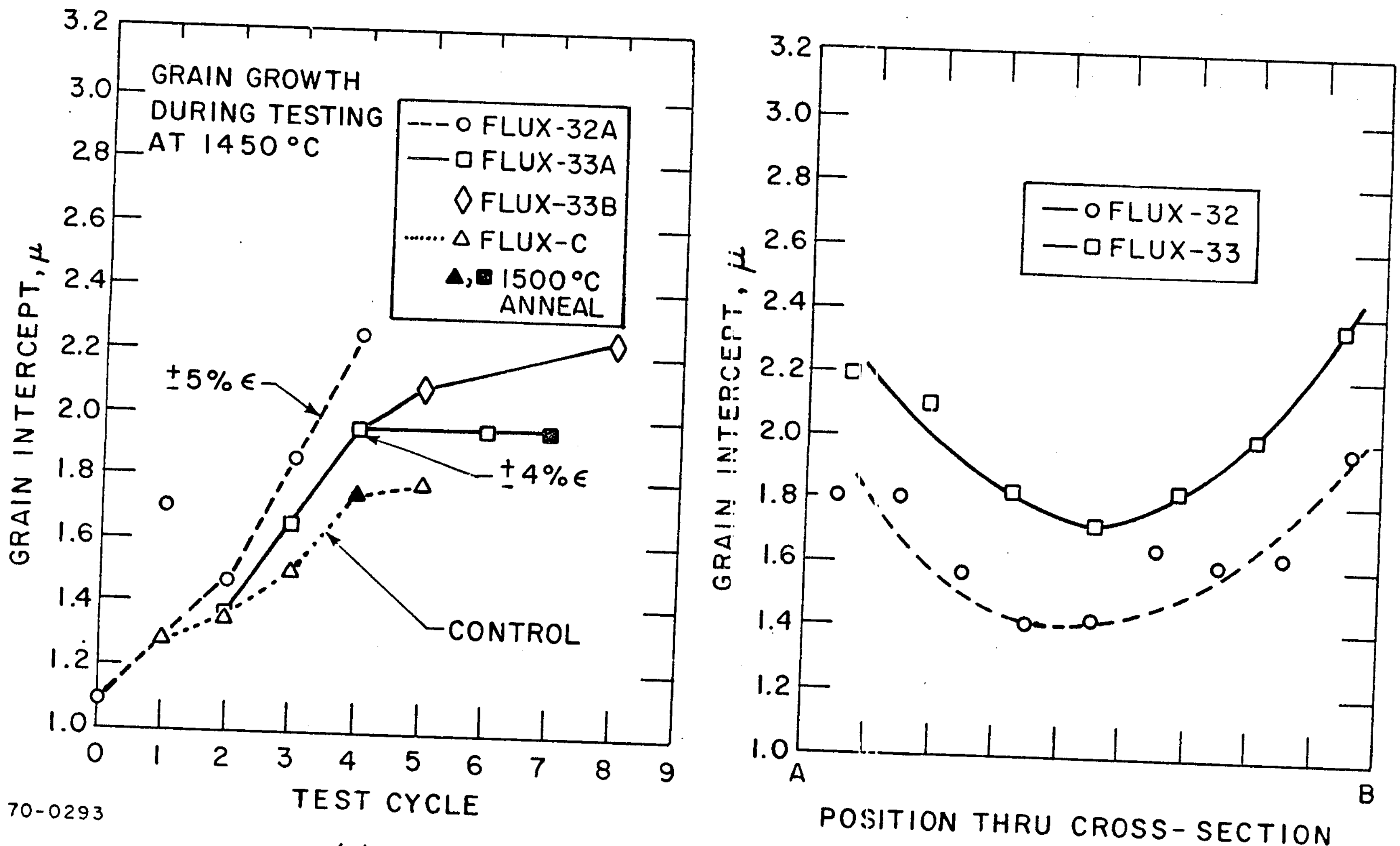
Figure 5. Ratio of Relative X-ray Intensity for Specimen JC-1474 Forged 38% Showing Strong Basal Texture.

This type of crystallographic texture has been reported previously for alumina forged at much higher temperatures[8] and is strong evidence of basal slip. The orientation of the 'c' axis parallel to the forging direction has also been reported for the hcp metals in which the basal slip system is the most easily activated.[9] These results indicate that even at 1450°C in fine-grained material, basal slip is a major contribution to deformation. It should be emphasized that this forging was done under conditions of relatively low temperature and strain rate where the highly rate-sensitive deformation has previously been attributed to grain boundary sliding or diffusional creep.[6,10-13]

In the previous report evidence was presented which indicated strain enhanced grain growth.[2] This was in the form of surface grain size measurements made after successive cycles in multiple bend tests for two different specimens and compared to a control specimen which accompanied the test bars through various test cycles to assess the effect of the thermal cycle. These data have some uncertainty associated with the possibilities of loss of dopant (MgO) from the surface, surface contamination from the furnace, and the effect of surface grain boundary interactions resulting in higher grain boundary mobility. In order to provide alternative evaluation, the two specimens were cross-sectioned, polished and etched and replicated. Photographs were taken at regular intervals through the cross-section on a traverse from one side to the other. The grain size was then measured for these and plotted as a function of position through the cross-section. The results are presented in Figure 6 along with the surface grain size results previously reported. These results indicate a minimum grain size at the center and an increase in grain size with distance out from the center. This is as expected if the grain growth is strain enhanced since the strain is approximately zero at the center and increases linearly to the surfaces. (Actually, the neutral axis tends to move toward the concave surface so that in multiple bending it moves back and forth past the center and there is no region of zero strain.) These combined results are taken as support for strain enhanced grain growth. This result is not surprising because the stress concentrations and boundary jogs resulting from grain boundary sliding must provide a strong driving force for boundary migration.

Although less attention has been given to fracture than flow mechanisms, it is equally important as it must be avoided for successful forging. Several observations regarding fracture have been made which are worthy of comment at this time. Intergranular separation has been previously reported to occur in alumina during deformation[6,11-13] and has also been observed here particularly after large strains (see Figure 4b). This is a particularly important problem, both in terms of eventually causing failure by intergranular fracture, but also because of the adverse effect upon subsequent properties.

The cause of cavitation in this material is not known with certainty; however, work in metal systems has indicated that it is directly dependent upon grain boundary sliding.[14] It should not be inferred from this, however, that cavitation is a necessary consequence of sliding, for it has been shown that considerable sliding may occur without cavitation resulting. The resistance to cavitation is presumably related to the ability of the material



(a) (b)

Figure 6. Evidence of Strain Enhanced Grain Growth. Part (a) Shows the Surface Grain Size Measured after Successive Tests in Two multiple Bend Specimens and for a Thermal Control Sample. Part (b) Shows a Plot of Grain Size Through the Cross-Section from Surface A to Surface B for These Two Specimens after Testing.

to relieve the stresses which develop at triple junctions and jogs. This relief in metals is thought to be limited largely by grain boundary migration;[15] however, it may be aggravated in ceramics and in particular, in alumina by the relatively high stresses necessary to activate non-basal slip. It has been shown in copper that the growth of boundary cavities, once formed, may be enhanced by the development of an internal pressure from entrapped gases.[16] Hot pressed alumina is known to have a problem of bloating on reheating due to condensation of gas so it is quite likely that this problem may frequently aggravate the cavitation problem.

Observations to date indicate that there are frequently patches which have a much higher than average density of intergranular cavities. An example of this shown in Figure 7. The cause of this high localized concentration is not known with certainty; however, it appears that many of the macroscopic cracks seen to date have had their origins in defective areas such as pore nests or in one material, large grain patches (see Figure 2).

The amount of cavitation observed does not depend only on the total amount of strain. The reversed bending specimens exhibit less boundary separation than seen in the 38% reduction forged specimen indicating, not surprisingly, that unidirectional straining is more serious. Examination of some specimens forged on a previous program[17] to 35 and 37% reduction at 1425°C revealed many essentially similar features to those described earlier for the forgings except that their was very much less intergranular separation as can be seen in Figure 8. Although these were forged at a higher rate, approximately 2×10^{-4} sec^{-1}, they had lower aspect ratios, L/D = 0.27, which results in significantly higher frictional constraint which effectively provides a hydrostatic pressure.[18] It is thought that the higher hydrostatic compressive stress is important in inhibiting cavitation.

More work in this area is warranted and is in progress. Two areas of interest have received particular attention. One is the effect of defects such as pore nests, high impurity sites and large-grained patches on causing early cracking. The second is attention to the problem of cavitation; one particular approach to this is to allow increased grain boundary mobility during testing to reduce the stress concentrations leading to cracking. The problem is that higher boundary mobility during testing to reduce the stress concentrations leading to cracking. The problem is that higher boundary mobility is usually accompanied by increased grain growth. One solution to this may be the use of two phase bodies, such as $Al_2O_3/MgAl_2O_4$ so that higher deformation temperatures can be used with less grain growth.

V. DISCUSSION

The work to date on flexural specimens and forged materials provides strong microstructural support for a combination of grain boundary sliding with at least part of the necessary grain shape change occurring by slip in fine-grained polycrystalline alumina even at low temperatures. The basal texture indicates that basal slip is the predominant slip system. Unequivocal evidence of non-basal slip must await transmission electron

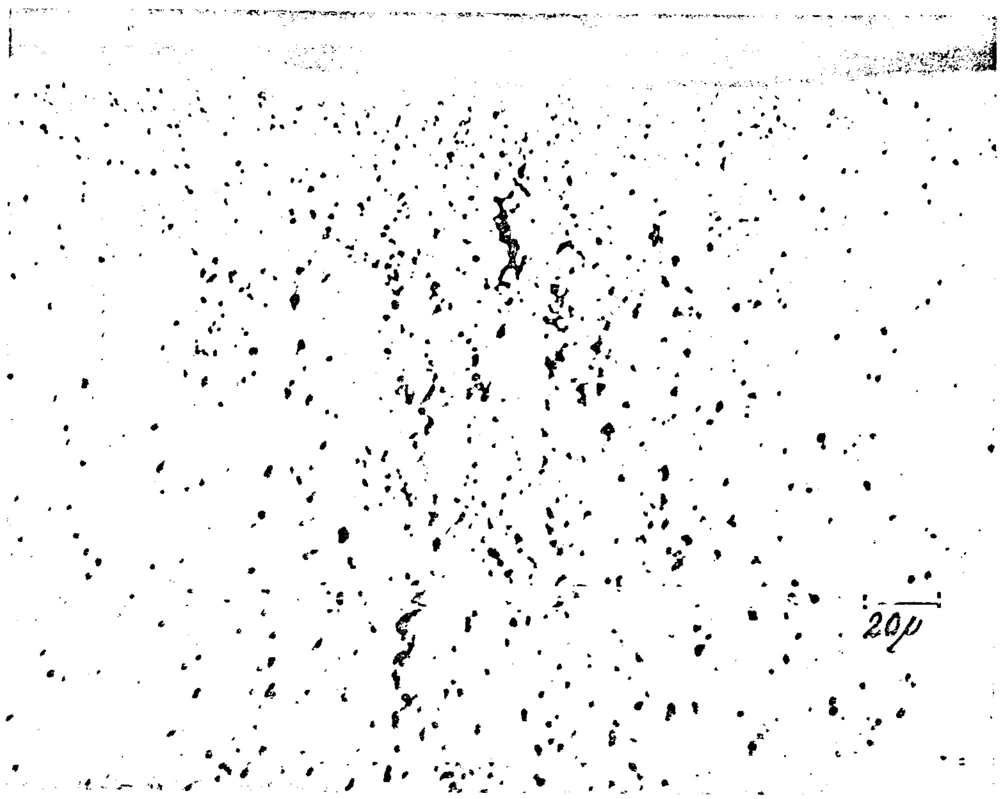


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Figure 7. Intergranular Cracking in Specimen FLUX-32.

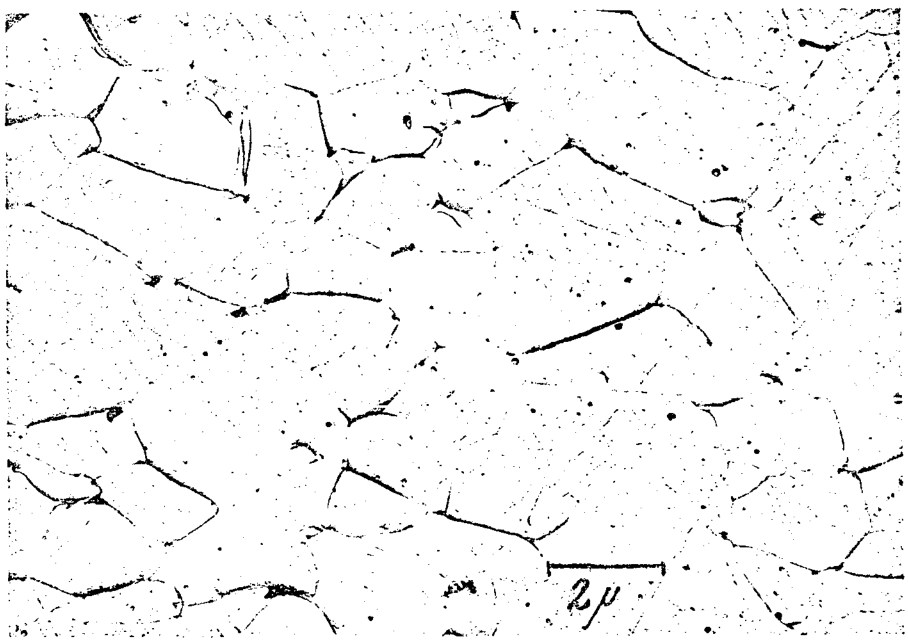

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Figure 8. Microstructure of Low Aspect Ratio Specimen, JC-11, Forged to 37% Reduction at 1425°C.

microscopy. It is thought likely, however, that grain boundary sources may make some non-basal slip possible. Microstructural evidence of diffusional creep is rather difficult to obtain so that any contribution from this mechanism is difficult to assess. It is possible that it may play a role in providing grain shape change.

The question of strain hardening effects cannot be assessed until the flexural test data have been reduced to true stress strain curves. However, some hardening from grain growth would be expected particularly in view of the evidence for strain enhanced growth. It may also be expected that boundary sliding would become more difficult as the boundaries become more distorted. The development of intergranular cracks, however, can be expected to result in a reduction of flow stress and may frequently offset any other effects.

VI. FUTURE WORK

During the next quarter, analysis of the multiple bend tests will be completed and testing of the grain size dependence specimens will be completed and the data analyzed. The torsion test facility should become available and will be used to provide more accurate flow stress data at higher strains. Microstructural evaluation, with particular attention to fracture mechanisms will be continued.

Several additional forging efforts are also planned. This will probably include another attempt at deep drawing a hemisphere and some compressive configurations.

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