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Attention: Mr. J. Gangler, Code RRM

Gentlemen:

Subject: Interim Technical Report No. 2
             Contract NASW-2187

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Very truly yours,

Allen A. Navarro
Contract Administrator

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Enclosures (11)

DEFORMATION PROCESSES IN FORGING CERAMICS

Progress Report No. 2

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FOREWORD

This work is being performed under the sponsorship of the NASA
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with Mr. J. Gangler as Project Monitor under Contract NASW-2187.

The work is being performed at the Avco Corporation, Systems Division,
Lowell, Massachusetts in the Materials Sciences Department, managed by
Dr. Thomas Vasilos. Mr. R.M. Cannon is directing the work with the assistance
of Dr. W.H. Rhodes. The authors wish to acknowledge the assistance of
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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 forging is being utilized
to investigate both the flow and fracture processes involved.

During this quarter, an additional hemisphere forging was
done which failed prematurely. Analysis and comparison with
available fracture data for Al_2O_3 indicated possible causes of
the failure. Examination of previous forgings indicated an
increase in grain boundary cavitation with increasing strain.

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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 necessary high temperature deformation 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 report of the first year of work included reviews of the work to date on 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. For alumina, retention of a relatively fine grain size is important even at very high temperatures in order to obtain adequate ductility. For polycrystalline magnesia at temperatures of 2100°C or above, adequate ductility from slip processes was indicated as probable.

During the first year's effort, extensive mechanical testing of fine-grained alumina was performed to clarify the contributing mechanisms of deformation at very fine grain sizes, to provide further data on flow stresses for use in forging, and to indicate the origins of cracking and cavitation at grain boundaries. In addition, a few forgings were done to correlate with the mechanical test results.

Considerable evidence was found for increasing contributions of non-Newtonian deformation processes, including grain boundary sliding and dislocation motion for grain sizes below 5 μ. In addition, some unexplained effects of specimen purity or test atmosphere on the flow stress were indicated.

Considerable capacity for deformation was demonstrated although several causes of cracking were indicated. For most of the flexural tests and the forgings, the limiting cracks occurred at defective areas in the specimens which included coarse grained patches, pore nests and regions of impurity concentration. In addition, cavitation at grain boundaries also develops and presumably would be the limiting feature if the defective regions were eliminated in the specimens. The appearance and growth of cracks was shown to be faster at higher strain rates with the associated higher stresses.

Further investigation of some of the unresolved problems is being undertaken during the present program. During this quarter, an additional Al_2O_3 forging was attempted which broke prematurely; this unanticipated fracture has not yet been satisfactorily explained. In order to provide a guide to allowable stresses and strain rates in forging, the high temperature fracture stress data from the literature and from several previous bend test studies have been collected and are discussed briefly in this report. Finally, further microstructural evaluation of the hemispheres forged in this program has been accomplished in order to further characterize the crack development.

II. FORGING

A hemisphere forging was done with the last blank of a previously made set. This blank was $Al_2O_3 + \frac{1}{4}\%$ MgO with a grain size of 3 μ, and had a 3-inch diameter. The graphite punch and die tooling, with a hemispherical radius of 1.05 inch, was the same as for the previous forging, D1600,[2] except that the die radius was increased because of cracking over the bend radius seen in the previous run. This run was made at an appreciably reduced strain rate in the initial stages. This was planned because the previous run had cracked early in the forging and because the earlier hemisphere, D1442, had some tearing at the apex; this tearing was not in the region of maximum thinning strain and so appeared to have been caused by excessive strain rate in the initial part of the run. The temperature for this forging was 1600°C, which is intermediate between the 1575°C used for the last forging which had cracked and the 1625°C used earlier on D1442 which had been more successful, but exhibited grain growth from 3 μ to about 7 μ. This trade-off is necessary in order to obtain as high a temperature as possible for maximum ductility and yet keep grain growth to a minimum to keep the flow stress as low as possible for a given strain rate.

The results were surprising in that the blank cracked very early in the run; the deflection was only about 0.04 inch compared to the blank thickness of 0.068 inch. The run was terminated in order to analyze the piece and try to establish the cause of premature failure. The forging was surprising in that fracture occurred rapidly and at a relatively low load. Further, the forging had been in progress for more than an hour and the deflection was significantly less than had been calculated or than the target which had been set on the basis of the strain distribution found in D1442 hemisphere and the target strain rate of 2×10^{-5} sec^{-1} maximum. For the failure load, an approximate calculation of the maximum bending stresses at the center of the blank gave 12 Ksi; the calculation does not include stretching stresses which should not be large at that stage of deflection or possible contact stresses near the punch. This value of fracture stress is lower than expected (see the next section for further discussion of fracture strength). One possible cause is that the stress is biaxial tension; at low temperatures, a reduction of fracture strength under biaxial loading has been reported for Al_2O_3[3]; however, the reduction was only 20%. At high temperature, a similar effect may be anticipated in the brittle regime and reduced fracture stresses for plastic tearing under biaxial loading are also probable.

The failure started at the center and propagated outward with several branches as seen in Figure 1. Examination of the fracture surfaces confirmed that the crack started in the center of the disc. However, it was surprising that the origin, as shown in Figure 2, appears to be at the surface on the punch side, which would be in compression from bending. In fact the plane of the crack was parallel to the scratches on the punch side left from surface grinding the blank. Crack initiation on the compressive side is not presently understood; even if stretching stresses were appreciable, the higher tensile stresses would still exist on the opposite side of the plate. This all suggests that contact stresses under the punch were important. One possible explanation is that the low deflection rate may have resulted from punch hang-up and that when the punch loosened, the immediate increase
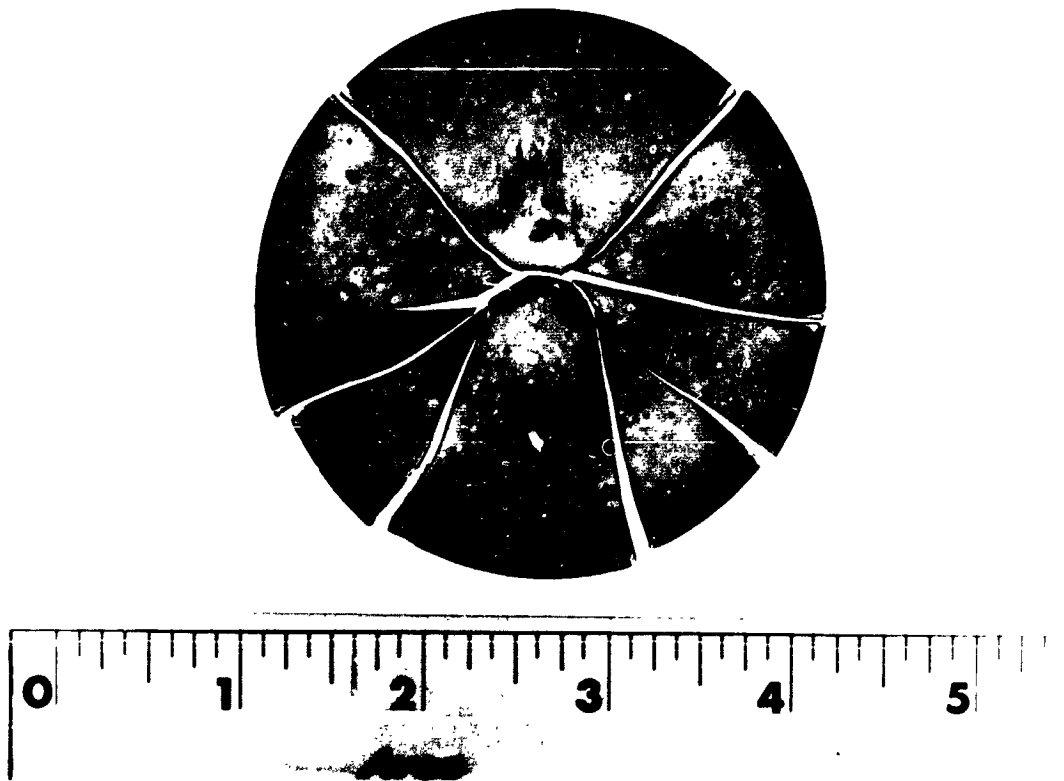
5603-1   1X
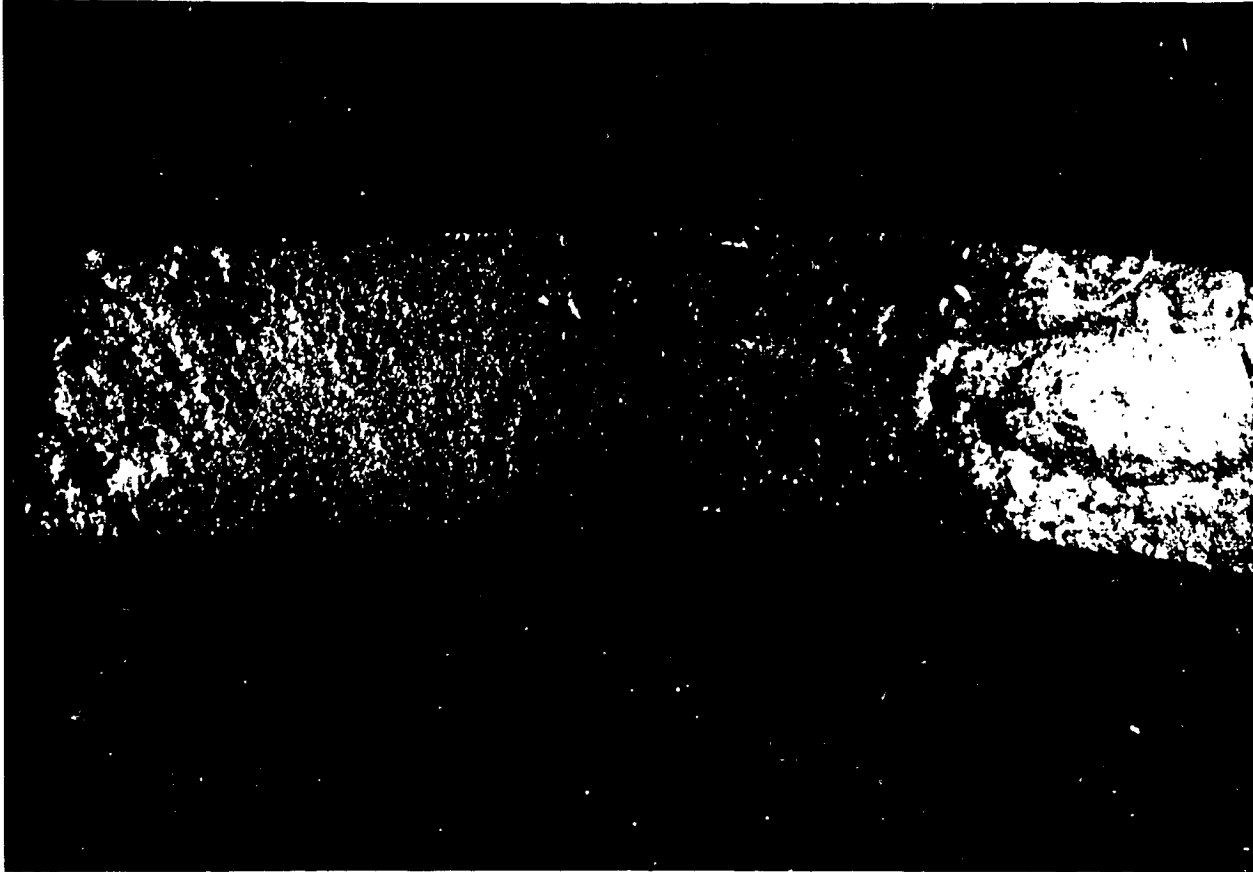
Figure 1. Premature Cracking in Hemisphere Run D1762. Most of the
           observable bending occurred after fracture.

5603-2   22X

Figure 2.   Fracture Surface of Crack Showing the Apparent Origin
            on the Surface Under the Punch.

in load was sufficient to initiate fracture. An alternative possibility is
that grain growth was greater than expected for the long forging time and
the possible strain rate was then significantly reduced. These possibilities
will not be resolved until microstructural examination of the piece is
complete.

III. HIGH TEMPERATURE FRACTURE

To successfully forge these rate sensitive ceramics, the applied loads
must be controlled to give the desired strain rates without developing
stresses high enough to cause fracture. In order to minimize grain growth
and to keep the forging time reasonably short, it is desirable to forge at
the highest rates possible without causing fracture. To indicate allowable
stresses over the temperature range of interest, approximately 1400°-1900°C,
the reported fracture stress versus temperature data for polycrystalline
Al_2O_3 has been collected and is plotted in Figure 3. Most of the studies
directly concerned with fracture were conducted at temperatures below the
range of interest here. The concern was primarily with short time fracture;
the lines from Spriggs, Mitchell and Vasilos[4] are typical of these results.
In order to supplement these data and to further indicate the effects of
plastic deformation, the data from three previous studies of flow stress-
strain rate were reviewed.[1,5,6] Most of these data are plotted as individual
points indicating either fracture or the highest flow stresses which were
measured in specimens where fracture did not occur. In some cases, these
highest stresses were well below the expected fracture stress for the strain
rates investigated. These data cover a range of grain sizes from 1 to 15 μ
and strains at failure as high as 3%. The line marked high purity represents
a similar compilation[7] of short time and plastic test results for a 1 μ,
high purity Al_2O_3. The data for C79 were the average of several tests at
fast enough strain rates to cause brittle fracture. Also plotted are the
highest flow stresses reported by Folweiler[8] in similar stress-strain rate
tests for material of 7 to 30 μ grain size. Fracture was reported to occur
at these highest points in this study.

As can be seen in Figure 3, these data indicate a rather broad range
of fracture stresses. This is not surprising considering the range of grain
sizes and strains before failure which are included. At high temperature,
the fracture behavior can be broken into two broad categories. At sufficiently
high stresses, the fracture is rapid and the fracture surfaces are brittle
in appearance. This behavior can qualitatively be considered in terms of a
Griffith fracture criterion, in the sense that rapid fracture occurs when
the flaw size becomes critical. Where plastic flow is occurring, the critical
flaws may develop as a result of growth of intrinsic flaws or grain boundary
separation before the rapid crack propagation stage; it is anticipated that
this may result in a reduction in fracture stress compared to that of the
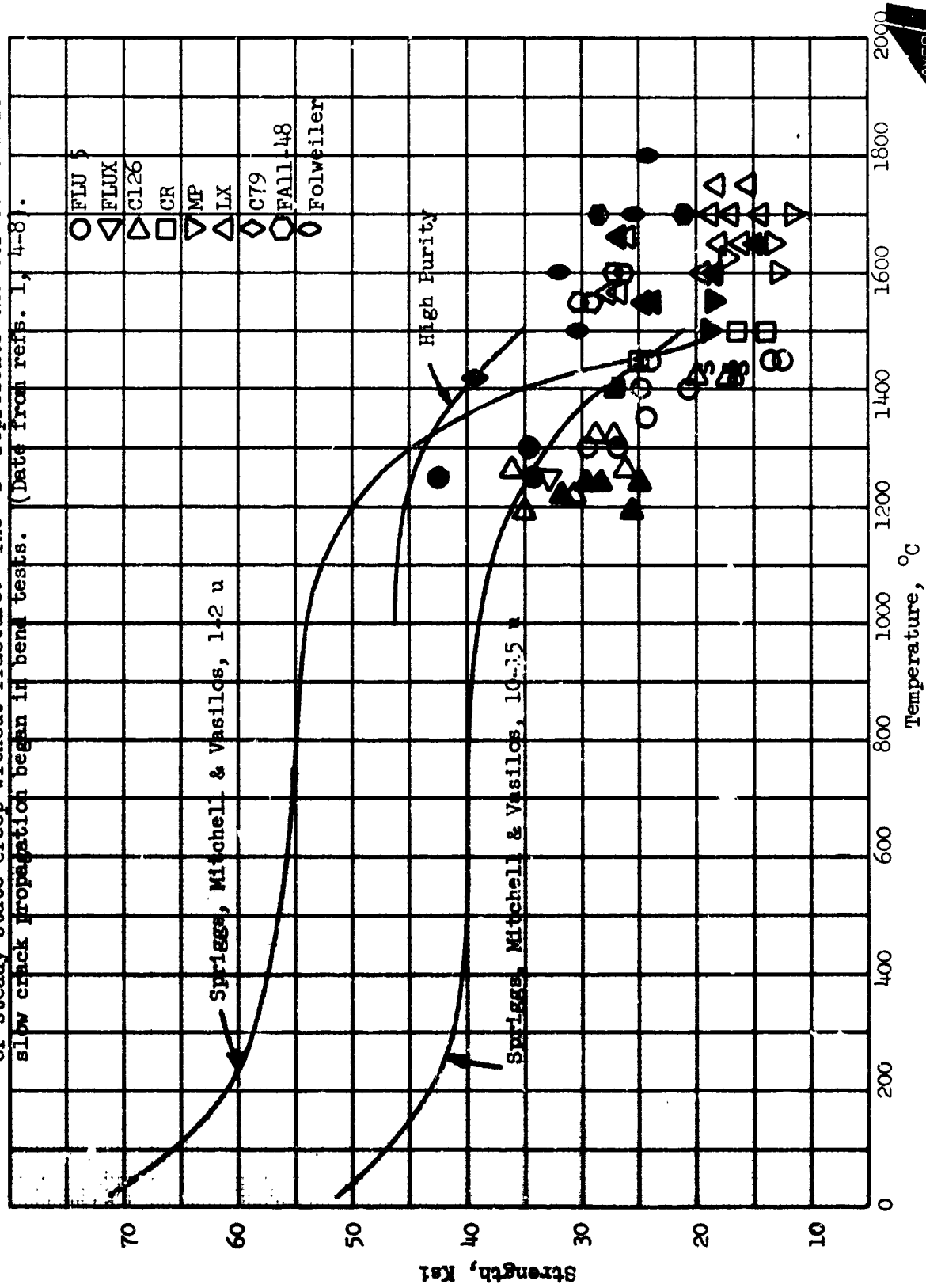unstrained material tested at higher rates.

A second mode of fracture is also seen where a significant amount of
stable crack growth occurs by tearing during plastic deformation. At lower
stresses or where stress gradients are high, this stable growth can be an
appreciable fraction of the cross-section before rapid fracture occurs.
This type of behavior was seen in the multiple bend tests reported last
year and in local tearing seen in forging.[1] At high temperatures, the
distinction between these two modes is partially qualitative since at

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Figure 3. Compilation of Fracture Stress at Temperature for Polycrystalline Al_2O_3. The full symbols represent actual fracture and the open symbols represent achievement of steady state-creep without fracture. The 's' represents the stress at which slow crack propagation began in bend tests. (Data from refs. 1, 4-8).

Legend:
- ○ FLJ 5
- ▽ FLJX
- △ C126
- □ CR
- ▽ MP
- ◁ LX
- ◇ C79
- ⬠ FAll-48
- ⬡ Folweiler

High Purity

Spriggs, Mitchell & Vasilos, 1-2 u

Spriggs, Mitchell & Vasilos, 10-15 u

Strength, Ksi

Temperature, °C

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intermediate rates the intrinsic flaws may be enlarged by a small amount of stable growth. In general, however, a significant difference exists in the fracture surface appearance between the two modes, and significant plastic flow can occur concurrent with the slow crack growth. At present, the quantitative stress, strain, and strain-rate requirements for stable crack growth are not known. The results from the multiple bend tests of last year are also shown on Figure 3; the stresses at which observable cracks developed are shown. These results suggested a strong stress dependence and showed that significantly higher strain could be obtained at lower strain rates, with the commensurately lower stresses, before crack growth became significant.

Comparing the nominal stress value of 12 Ksi, which was calculated for the hemisphere forging, D1762, previously discussed, it can be seen that this value is somewhat low, but not totally unreasonable. It should be noted, however, that the fracture occurred at a very low strain; the maximum bending strain was calculated to be about 0.2% when failure occurred. This value is then somewhat low for brittle fracture to occur even considering that the stress is biaxial.

IV. MICROSTRUCTURAL EVALUATION

Further examination of polished and polished and etched cross-sections of the previous hemispheres, D1442 and D1600, was completed. Although i was found, as reported earlier[2], that coarse grained patches existed in these pieces, and that they frequently caused tearing, cross-sections of material away from these were examined by replication and electron microscopy to determine the effects of the strain. A core of hemisphere D1442 was taken near the apex in the region of greatest thinning strain. This area had experienced an average biaxial bend strain of 3.9% and a thinning strain of 9.9%. Using the usual equivalent strain formula:

$$\bar{\epsilon}^p = \sqrt{\frac{2}{9}\left[(\epsilon_1^p - \epsilon_2^p) + (\epsilon_2^p - \epsilon_3^p) + (\epsilon_3^p - \epsilon_1^p)\right]}$$

these can be combined to find the total equivalent strain which linearly varies from 2.2% at the I.D. where the bending and thinning strains cancel, to a maximum of 17.6% at the O.D. These values are approximate in the sense that they only represent the final average strains and do not account for any redundant strains in the actual deformation.

Micrographs are shown in Figures 4, 5, and 6 of typical regions in the cross-section near the O.D., from the center, and near the I.D., providing a scan along the strain gradient. These show an increase in the amount of grain boundary cavitation with the increase in strain. The difficulty in assessing the actual amount of cavitation can be appreciated by comparing the as-polished and the etched views. It is obvious that etching significantly increases the apparent amount of porosity. It is not certain, however, whether this occurs because the triple points have porosity which is not apparent in the as-polished condition, or simply because of other effects such as higher residual strains at the points of constraint to grain boundary sliding. Most of the cavitation is at triple points rather than along grain faces. Areas from D1600 in the regions of high strain were similar in nature to these micrographs.

72297   (a)   1500X

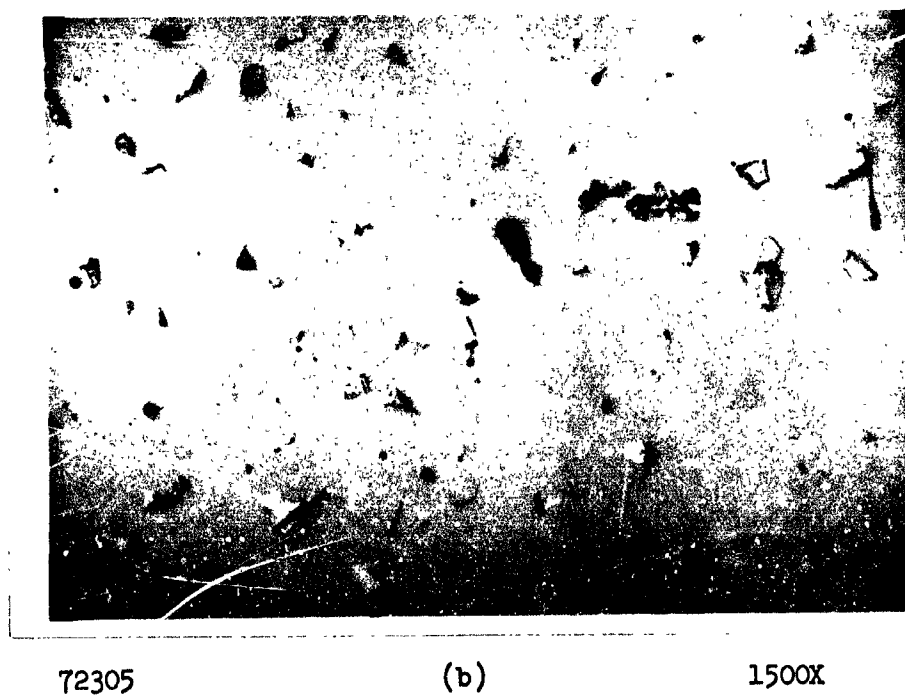72305   (b)   1500X
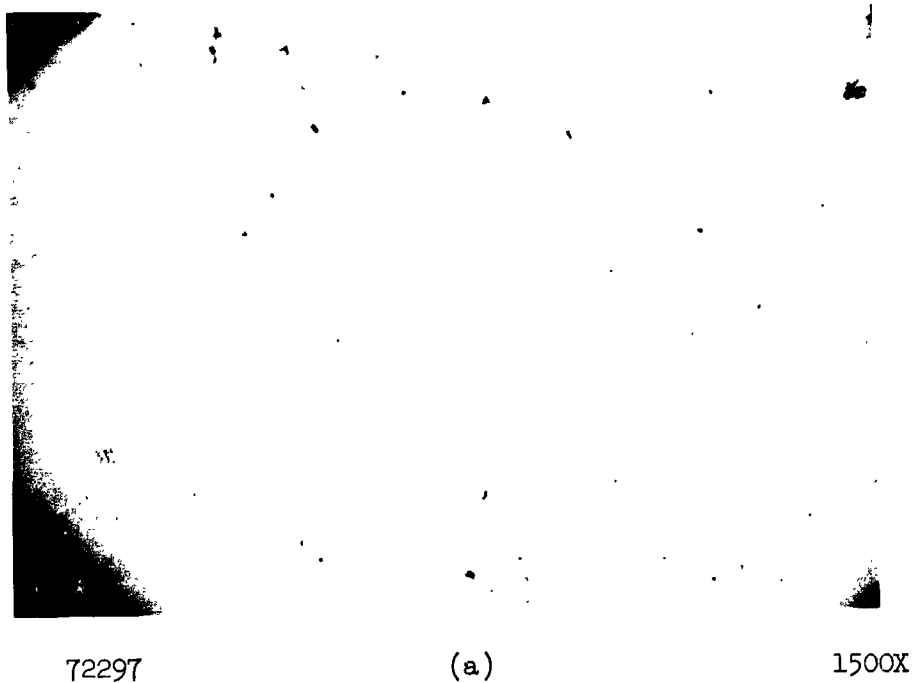
Figure 4.   Cross-Section of Hemisphere D1442 Near the O.D. in the (a)
Polished, and (b) Polished and Etched Condition.

72296   (a)   1500X

72307   (b)   1500X

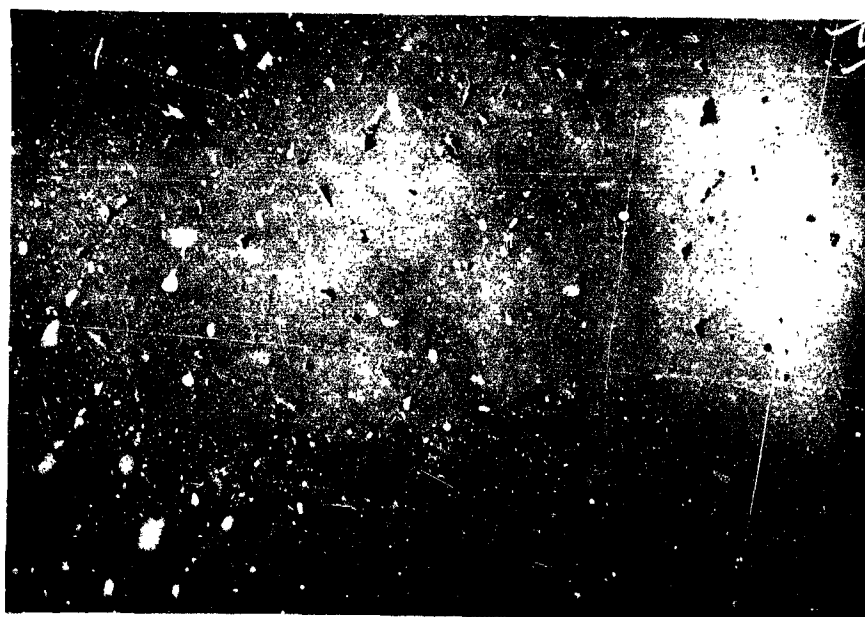Figure 5.   Cross-Section of Hemisphere D1442 from the Center Region in the (a) Polished and (b) Polished and Etched Condition.

72295   (a)   1500X

72306   (b)   1500X

Figure 6.   Cross-Section of Hemisphere D1442 Near the I.D. in the (a) Polished, and (b) Polished and Etched condition.

In this forging the visible tearing was restricted to the apex region, to regions adjacent to coarse grained patches, and to the I.D. surface where a layer of severe grain growth occurred. As discussed, the apex tearing is thought to have been caused by excessive strain rate during the early part of the forging. The tearing near coarse grained particles occurred as a result of the strain concentration around these "hard" regions. The grain growth on the I.D. apparently resulted due to interaction with the graphite lubrication used on the punch; this problem has been subsequently eliminated by changing the punch coating. By eliminating these three specific problems, the grain boundary cavitation would then become the limiting problem. A good example of how these cracks grow and eventually connect to form cracks is shown in Figure 7, which is from a large grained region near the surface. This emphasizes the importance of maintaining a fine grain size which reduces the strain concentrations from boundary sliding as well as the applied stresses.

V. FUTURE WORK

During the next quarter, the stress-strain rate tests reported during the last quarter will be finished and analyzed to assess the change in strain rate sensitivity which was found; these results will be combined with microstructural examination to indicate the cause of this behavior.

Further hemisphere forgings are also planned. Further modifications will be made to eliminate the early cracking.

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72299   3000X

Figure 7. Development of crack from growth and connection
           of triple point and grain boundary cavities; in
           large grain region near the surface of Hemisphere
           D1442.