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EFFECT OF STRESS ON PERMEATION THROUGH ALUMINA MEMBRANES

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Publication Date
1964-04-01
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Berkeley, California
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Amin Edwards Atallah
(M. S. Thesis)

April 1, 1964
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EFFECT OF STRESS ON PERMEATION THROUGH ALUMINA MEMBRANES

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April 1, 1964

ABSTRACT

The effect of biaxial tensile stress on gas permeation through normally impermeable alumina membranes was investigated at room temperature.

Tests were conducted at various loading cycles on two groups of alumina bodies. Group A had a membrane thickness ranging between 700 and 1000 microns. Group B had a membrane thickness of approximately 125 microns. The load and the length of time of load application were independently varied.

No permeation could be detected through the thick alumina membranes. Thin membranes showed no permeation under low loads; leakage through the sealing cement prevented detection of permeation through these membranes at high loads.

Heating in air was found to heal microcracks induced by diamond-grinding the thin alumina membranes. It is proposed that vacuum heating to reactivate membranes which previously exhibited permeation had also healed previously induced microcracks.
I. INTRODUCTION

Impermeable ceramics capable of prolonged use at high temperatures and under stress are required for several applications. Ceramic materials impermeable to gases are, for example, used as envelopes for electron tubes.

Robertset al. investigated the effect of temperature on diffusion of oxygen, nitrogen, and inert gas through alumina tubes. They found that sintered alumina materials which are impermeable to oxygen, nitrogen, and inert gases at room temperature generally retain their impermeability up to temperatures near $1500^\circ C$. These gases then started diffusing at a rate that increased approximately exponentially with increasing temperature. The stress imposed on the ceramic in these studies was negligible.

Stansfield conducted similar experiments on commercially manufactured high-alumina tubes at various temperatures and tensile stress levels. His specimens were divided into two groups according to wall thickness. The first group has a 1/4-in. wall thickness. The second group had a reduced section of about 0.025-in. wall thickness which was centered in the constant-temperature zone. The tests on the first group were conducted at temperatures ranging between 25 and $270^\circ C$ and at cyclical tensile stress levels ranging between 2000 and 7700 psi. The tests on the second group were conducted at temperatures varying between 714 and $963^\circ C$ and at cyclical tensile stress levels ranging between 3500 and 12,000 psi. Stansfield observed no permeation through the alumina specimens in either group.

The only direct experimental evidence of stress-induced gaseous permeation at room temperature was obtained by Studt. He conducted his studies on four commercially manufactured high-alumina disks with alumina content varying from 95% to 99.9% and with bulk porosity varying from 0.4% to 7.2%. Studt found that only two bodies, which at room temperature were normally impermeable, became permeable under stress. This stress-dependent permeability was shown to be a function of the microstructure of the material. About 5% closed porosity was
found necessary for stress-enhanced permeation. The burst, which was a rapid, spontaneous flow of helium, was suggested by Studt to be only through pores interconnected by microcracks. This burst decreased and finally vanished upon continuous application of cyclic stress at a constant stress level (Fig. 1). Increasing the stress level again initiated a flow with the same gradual reduction. Studt postulated that adsorption of gas atoms on the surface of microcracks eventually plugged channels and thus decreased the flow even under cyclic stress conditions.

In this study four disks previously used by Studt—namely, (1-2), (1-7), (1-8), and (1-9), and three new ones, A1, A2, and B2 of the same composition and manufactured by the same firm—were selected for investigation in an attempt to duplicate Studt's results.
Fig. 1. Peak flow rate for applied and released tensile stress as observed by Studt (reference 3) on Specimen 1-7.
II. EXPERIMENTAL PROCEDURES

A. Specimen Preparation

The disks were tested in a manner such that a small membrane was subjected to a biaxial tensile stress. The disks were mounted in the testing device as fixed-edge rigid diaphragms, as shown in Figs. 2 and 3. A ring load was applied normal to the surface of the disks. Blind holes were drilled in the disk inside the area encompassed by the ring load. Figure 4 shows the two blind hole geometries used. Having the hole drilled past the neutral axis of the disk will create a membrane that, when loaded, is subjected to biaxial tensile stress (Fig. 5). In Fig. 6 (upper) the geometry for one 1/4-in.-diameter hole is shown; and in Fig. 6 (lower), for four 1/8-in.-diameter holes.

Two of the new specimens, A1 and B2, and four specimens on which Studt detected permeation were selected for Group A. All specimens were in the form of disks 1/8 in. thick by 3 in. in diameter with membrane thickness varying from 700 to 1000 microns (Fig. 6, upper). They were impermeable to helium under stress-free conditions.

One new disk, A2, and one of Studt's disks were then chosen for Group B. The surface opposite the blind holes was ground on a diamond wheel until a membrane thickness of approximately 125 microns was reached (Fig. 6, lower).

B. Thermal Treatment

The disks of Group A, previously used by Studt, were heated for 12 hours after each run in a vacuum of $10^{-3}$ to $10^{-4}$ mm of Hg and at a temperature of 1200° C in an attempt to eliminate the effect of adsorption of helium atoms on the surface of the microcracks. This was an attempt to reactivate those disks previously exposed to helium while being stressed; any helium atoms that plugged the microcracks would be expected to be removed at this pressure and temperature.

The two disks of Group B, which had been ground to reduce the membrane thickness, were heated in air for 2 hours at various
Fig. 2. Schematic representation of disk-stressing device, Group A.
Fig. 3. Schematic representation of disk-stressing device, Group B.
Fig. 4. Schematic indication of locations of blind holes in fabricated specimens.
Fig. 5. Cross section of disk, showing membrane position before and after application of load.
Fig. 6. Schematic cross section of the disks: (upper) before reducing the thickness of the membrane (700 to 1000 μ); (lower) after grinding the surface opposite the blind hole to reduce the thickness of the membrane (≈ 125 μ).
temperatures ranging between 600° C and 1500° C in an attempt to heal the microcracks induced by grinding. The process might be due to the diffusion of the glass present in the body, or to atomistic diffusion, which would tend to eliminate the induced microcracks.

C. Equipment

Mechanical tensile stresses were induced in the membranes by concentrically loading fixed-edge alumina disks (Figs. 2 and 3).

A mass spectrometer, sensitive to minute traces of helium, was used to detect flow through the alumina membranes. The mass spectrometer output was continuously recorded to allow observation of any rapid changes that might accompany the cyclic stresses. Helium, because of its relatively small size and its ability to act like a free atom during permeation, was used as the diffusing gas in all the experiments. All experiments were conducted at room temperature.

The apparatus previously used and described by Studt	extsuperscript{3} was modified. It was made more sensitive by reducing the volume of the collection chamber monitored by the mass spectrometer. The new collection chamber was directly attached by epoxy cement to the alumina disk. This attempted to eliminate the effect of any possible leakage through the O rings "a" and "b" (Figs. 2 and 3).

The apparatus consisted of four chambers (1, 2, 3, and 4, in Fig. 2). The alumina disk was a rigid diaphragm in the loading device. The four chambers were evacuated to $10^{-5}$ mm of Hg, and maintained at this pressure for approximately 12 hours.

Ram loading was used. Loads were applied to the ram by means of a diaphragm cylinder actuated by air pressure which was controlled by a pressure regulator.

The length of time of load application was controlled by an automatic timer. The time of load application and stress-free condition could be varied independently.

The pressure regulator was calibrated by Studt in terms of (a) pressure at the diaphragm cylinder, and (b) micrometer settings
of pressure regulator. Calibration was performed with SR-4 strain gauges on an aluminum specimen which was previously calibrated in terms of total load on the specimen vs strain. Strain was measured directly with a Baldwin SR-4 strain-gauge indicator and converted to load from the calibration curves of the aluminum strain gauge device. Load vs regulated pressure gauge readings are reproduced in Fig. 7.

D. Microstructure

Sections, diamond-sawed from the new disks, were ground successively with 400, 600, and 1000 mesh silicon carbide. The sections were polished on a vibrating lapping machine with 2- to 4-micron diamond paste for 5 hours. Orthophosphoric acid was used to etch the specimens. Etching was achieved in 5 min at temperatures between 190 and 200° C.

The polished and etched surfaces were covered by a very thin layer of gold by vaporization in vacuum. This prevented reflection from beneath the surface of the sample. Photomicrographs of the cross-section of the disks were then taken. Figure 8a shows a representative structure.

The grain size of the new alumina disks varied from 5 to 30 microns. The pores appeared at junctions of grain boundaries and in the interior of individual grains.

The microstructure of the new disks differed from those reported by Studt for the same manufacturer's designation. Apparently in the time intervening between Studt's work and this investigation the manufacturer had changed his processing to give larger-grained bodies with different pore distribution from the original body. Studt showed grain sizes in the range 3 to 5 microns with pores only at grain boundaries. Figure 8b shows a representative structure of the specimen used by Studt.
Fig. 7. Load applied as a function of regulated pressure.
Fig. 8. Photomicrographs showing grain and pore structure of (a) new disks, (b) alumina disks used by Studt (reference 3).
E. Test Procedure

Two different procedures were used to test the specimens. For Group A the following steps were taken (Refer to Fig. 2);

1. The mass spectrometer output was connected to a chart recorder.
2. The disk was placed in the loading device with valves B and E closed and A, C, D, F, and G opened. The four chambers were then evacuated to $10^{-5}$ mm of Hg and maintained at this pressure for approximately 12 hours.
3. One atmosphere of helium pressure was established in chamber 2 by closing valve A and opening valve B.
4. Cyclic loads in the range of 100 to 700 lb were then applied for various cycle times ranging from 30 sec to 5 min.
5. Chamber 2 was then evacuated. The epoxy cement was tested by introducing helium in chamber 3 and monitoring chamber 4. Chambers 1 and 2 were evacuated by pump 2.
6. Chamber 3 was evacuated, helium was again introduced in chamber 2. Valve C was closed and valve E opened, connecting chamber 3 to pump 1 and leaving only chamber 4 connected to the leak detector. Chamber 1 was evacuated by pump 2.
7. Step 4 was repeated.
8. The disk was then disconnected from the apparatus and heated to 1200° C in a vacuum of approximately $10^{-3}$ mm of Hg for 12 hours.
9. The whole procedure was again repeated.

For Group B a slight change in the connection of the valves was made. Chambers 1, 2, and 3 were evacuated by the same vacuum system. A schematic representation is shown in Fig. 3.

1. The disk was ground on a diamond lap until a membrane thickness of 125 microns was reached.
2. The disk was heated in air to about 1200° C to heal any micro-cracks induced by grinding.
3. The disk was placed in the loading device with valve B closed and valves A, D, F, G, and C opened.
4. One atmosphere of helium pressure was established and kept at constant pressure in chamber 2 by closing valve A and opening valve B.

5. Cyclic loads in the range of 25 to 400 lb were applied for various cycle times varying from 30 sec to 5 min.

6. The mass spectrometer output was continuously recorded.
III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Observations on Group A Disks

Group A, the set of disks with membrane thicknesses from 700 to 1000 microns, included four disks previously used by Studt\textsuperscript{3} and two new disks. Studt had observed that after the disks were cycled in an atmosphere of helium the intensity of helium bursts on application or release of load decreased. Therefore, those disks that had been subjected to helium were tested and then heated in a vacuum below $10^{-3}$ mm of Hg at 1200° C for 12 hours in an attempt to remove any helium absorbed on the surface or in microcracks.

The first disk tested (Table I), in the group of disks studied by Studt, had four 1/8-in. blind holes, as shown in Fig. 4b. The first run made before any vacuum heat treatment indicated that the O rings "a" and "b" in Fig. 2 leaked helium when a load was applied to the disk. The first indication of helium leakage was detected by the ionization-type vacuum gauge monitoring the vacuum in chamber 1. On load application the pressure in chamber 1 would immediately rise from approximately $10^{-5}$ mm of Hg to more than $10^{-2}$ mm of Hg. The vacuum would then be recovered slowly while the load was applied. On release of load a pressure rise of much smaller magnitude would be observed. This observation proved that under the application or release of load, O ring "a" in Fig. 2 would allow helium leakage.

Because chamber 3 was connected to a separate pumping system, the indication of helium leakage was not observed as a pressure rise on the ionization gauge monitoring that system. However, when chambers 3 and 4 were both connected to the leak detector, an erratic burst of helium was detected on the application or release of load. Therefore, O ring "b" in Fig. 2 was also leaking, resulting in helium's being present in chamber 3 but at very reduced pressures. When the membranes were monitored alone (chamber 3 to pumping system 1 and chamber 1 to pumping system 2) no detectable permeation or bursts were observed under cyclic loading.
Table 1. Study 1-7 of Studt's disk; geometry as in Fig. 4b. Membrane 700 to 1000 μ thick.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>System</th>
<th>Treatment</th>
<th>Experimental procedure and results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>None</td>
<td>Pressure applied on the disk from 5 to 17.5 psig in 2.5-psig increments.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cycle time 30 sec on, 30 sec off</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>O rings leak on application and release of pressure.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Pressure-dependent diffusion of helium through epoxy.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>No diffusion or helium burst through membrane.</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>Heated for 12 hrs at 1200° C and approx 10⁻⁴ mm Hg.</td>
<td>Same as in run 1</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>As in run 2</td>
<td>Pressure applied on the disk from 7.5 to 35 psig in 2.5-psig increments.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cycle time 15 sec on, 15 sec off, and 30 sec on, 30 sec off.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>O rings leak on application and release of pressure.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Pressure-dependent diffusion of helium through epoxy.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>No diffusion or helium burst through membrane.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Failure of membrane at 35 psig.</td>
</tr>
</tbody>
</table>
The epoxy seal between the copper ring attached to the bellows and the alumina did not leak helium until very high loads (approximately 300 to 400 lb) were applied to the disks. Under these loads, introducing 1 atmosphere of helium into chamber 3 caused a detectable rise in the helium pressure in chamber 4. The flow was extremely small but detectable.

The system, when operated with chamber 1 to pumping system 2, chamber 3 to pumping system 1, and chamber 4 to the leak detector, was insensitive to the leakage of the O rings and epoxy. Operating under these conditions permitted monitoring of helium permeation through the membrane area alone. No detectable helium permeation was observed in any of the four disks previously investigated by Studt either prior to or after a vacuum heat treatment (see Tables I, II, III, and IV).

The two new disks with membrane thicknesses between 700 and 1000 microns did not show O-ring leakage. Therefore, the epoxy leakage was not checked. Under identical testing conditions, as outlined above, these disks did not show any detectable helium permeation under load, or any bursts on application or release of load (Tables V and VI).

B. Observations on Group B Disks

It was decided to test thinner membranes because of the possibility of (a) reduced permeation of thick membranes under stress, (b) the plugging of Studt's disk by helium which was not removed by a vacuum heat treatment, and (c) the difference in microstructure observed in the new disks in comparison with those shown by Studt. The disks with blind holes, 700 to 1000 microns thick, were surface ground with diamond abrasive on the side opposite the hole. The membranes were reduced to approximately 125 microns thickness in this manner. One of Studt's disks and one new disk (Tables VII and VIII) were tested with reduced membranes. The diamond grinding induced microcracks in the membrane, which caused previously vacuumtight membranes to permit free permeation of helium. No visual evidence of these cracks
Table II. Study 1-8 of Studt's disk; geometry as in Fig. 4b.
Membrane 1000 \( \mu \) thick.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>System</th>
<th>Treatment</th>
<th>Experimental procedure and results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>Heated for 12 hrs at 1200° C and approx. ( 10^{-4} ) mm Hg.</td>
<td>Pressure applied on the disk from 5 to 20 psig in 2.5-psig increments. Cycle time 30 sec on, 30 sec off. O rings leak on application and release of load. Pressure-dependent diffusion of helium through epoxy. No diffusion or burst of helium detected through membrane.</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>Same as in run 1</td>
<td>Same as in run 1.</td>
</tr>
</tbody>
</table>

Table III. Study 1-9 of Studt's disk; geometry as in Fig. 4b.
Membrane 998 \( \mu \) thick.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>System</th>
<th>Treatment</th>
<th>Experimental procedure and results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>None</td>
<td>Pressure applied on the disk from 5 to 12.5 psig in 2.5-psig increments. Cycle time 15 sec on, 15 sec off. No diffusion or burst of helium detected through membrane.</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>Heated for 12 hrs at 1200° C and approx ( 10^{-4} ) mm Hg.</td>
<td>Pressure applied on the disk from 5 to 10 psig in 2.5-psig increments. Cycle time 15 sec on, 15 sec off. Pressure-dependent diffusion of helium through epoxy.</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>Same as in run 2</td>
<td>Pressure applied on the disk from 5 to 20 psig in 2.5-psig increments. Cycle time 30 sec on, 30 sec off. O rings leak on application and release of load. No diffusion or helium burst detected through membrane.</td>
</tr>
</tbody>
</table>
Table IV. Study 1-2 of Studt's disk; geometry as in Fig. 4a.
Membrane 700 to 1000 μ thick.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>System</th>
<th>Treatment</th>
<th>Experimental procedure and results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>Heated for 24 hr at 1200° C and approx 10^{-4} mm Hg.</td>
<td>Pressure applied on the disk from 10 to 20 psig in 2.5-psig increments. Cycle time 30 sec on, 30 sec off. O rings leak on application and release of pressure. Pressure-dependent diffusion of helium through epoxy. No diffusion or burst of helium through membrane.</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>Heated for 12 hrs at 1200° C and approx 10^{-4} mm Hg.</td>
<td>Pressure applied on the disk from 5 to 20 psig in 2.5-psig increments. Cycle time 30 sec on 30 sec off. Same results as in run 1.</td>
</tr>
</tbody>
</table>

Table V. Study A-1 of new disk; geometry as in Fig. 4a.
Membrane 889 μ thick.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>System</th>
<th>Treatment</th>
<th>Experimental procedure and results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>None</td>
<td>Pressure applied on the disk from 10 to 22.5 psig in 2.5-psig increments. Cycle time 30 sec on, 30 sec off. No leak through O rings. No diffusion or burst of helium through membrane.</td>
</tr>
</tbody>
</table>
Table VI. Study B-2 of new disk; geometry as in Fig. 4b. Membrane 787 μ thick.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>System</th>
<th>Treatment</th>
<th>Experimental procedure and results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>None</td>
<td>Pressure applied on the disk from 15 to 25 psig in 2.5-psig increments. Cycle time 30 sec on, 30 sec off. No leakage through O rings. No diffusion or burst of helium through the membrane.</td>
</tr>
</tbody>
</table>

Table VII. Study 1-2 of Studt's disk; geometry as in Fig. 4a. Membrane 127 μ thick.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>System</th>
<th>Treatment</th>
<th>Experimental procedure and results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>Surface ground on a diamond wheel.</td>
<td>Outshooting the lowest scale when 1 atmosphere of helium was established in chamber 2. Presence of microcracks.</td>
</tr>
<tr>
<td>2</td>
<td>2</td>
<td>Heated in air for 2 hr to 600°C.</td>
<td>Microcracks still exist.</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>Heated in air for 2 hr to 1000°C</td>
<td>Microcracks still exist but the amount of flow could be detected on the lowest scale.</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>Heated in air for 2 hr to 1200°C</td>
<td>Microcracks completely healed.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Pressure applied on the disk from 3 to 11 psig in 1-psig increments.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cycle time 5 min on, 5 min off.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>O rings leak on application and release of pressure.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Steplike jumps were detected on applying and releasing stress before sample broke at the maximum pressure.</td>
</tr>
</tbody>
</table>
C. Discussion of the Results

The results obtained by Studt and those obtained in this investigation do not agree. The disagreement may be attributed to the difference in sample preparation and operating procedure.

The disks used by Studt were first fired, then blind holes to form the membranes were drilled either with a diamond bit or with B₄C powder abrasive in an ultrasonic grinder. The membranes were relatively thick compared with the average grain diameter of the specimen. The disks that showed helium permeation were those with higher impurity content and containing residual closed-pore porosity. The higher impurity content is more conducive to the formation of a glassy phase along grain boundaries, as shown in Fig. 8b.

In this investigation it was shown that diamond grinding induced microcracks in both a disk used by Studt and a new disk. The diamond grinding performed by Studt may also have induced microcracks in the membranes containing glassy phase; the disks having lower impurity content are less likely to crack during diamond grinding and, therefore, do not display the bursting phenomenon.

In this investigation the four disks used by Studt were tested prior to any heat treatment and did not show helium permeation. There results agree with the conclusion of Studt that the bursting phenomena could be suppressed after simultaneous exposure to helium and stress. The subsequent heat treatment, in an attempt to reactivate the membranes, was later shown to be sufficient to completely heal microcracks induced by diamond grinding. The disk used by Studt, in which microcracks were induced by surface grinding, was made vacuum tight at the same temperature but in a shorter time than used for reactivation of the thick membranes.

The grinding of the new disks to form the membrane was done before the disks were fired, consequently no microcracks were present and no helium permeation was observed. In the new disk that was surface-ground to form the thin membrane channel, flow of helium was observed, indicating the presence of microcracks, whereas nothing was observed before grinding.
The new disk required greater heat treatment to heal the micro-cracks than Studt's disk, this is most probably due to the much larger average grain size of the new disk and different microstructure (see Fig. 8).
IV. CONCLUSIONS

The possibility of vacuumtight ceramic bodies allowing permeation of helium when stressed at room temperature has not been confirmed or denied. Alumina bodies which had been observed to give bursts of helium on the application or release of load did not show this behavior in this investigation. Heating to 1200° C in vacuum did not reactivate the disks to give the burst phenomenon.

Thin membranes, 125 microns thick, did not give bursts of helium on application or release of loads up to 300 lb, or show any permeation under low loads. At high loads, above 300 lb, the leakage of the epoxy used in the seal and the O-ring seals could account for the bursts observed.

Heating in air can heal microcracks induced in ceramic materials by diamond grinding. The exact time and temperature needed to cause this recovery of vacuum tightness is dependent upon the microstructure and previous processing of the body.
ACKNOWLEDGMENTS

The author gratefully appreciates the counsel and direction of Professor R. M. Fulrath during the course of this investigation.

He is grateful to Mr. Robert Buehrig, who prepared the photomicrographs, and to Mr. Stephen Firestone, for the maintenance of the equipment and experimental assistance.

Finally the author acknowledges the helpful discussions and suggestions of Mr. Ronald Rossi and Mr. Orlin Stansfield.

This work was done under the auspices of the U. S. Atomic Energy Commission.
REFERENCES


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