COMPOSITE INORGANIC RESILIENT SEAL MATERIALS

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This report covers the period of work from December 1958 through December 1959.
The principal objective of this research program is to investigate and develop new concepts for novel and unconventional material combinations which would have resilience, recovery, strength, and chemical resistance at temperatures up to 1000°F. Major emphasis was given to configurations that would enable these materials to be used as static and dynamic reciprocating shaft seals.

Composite material combinations consisting of stainless steel and molybdenum fibers impregnated with tin, indium, magnesium, silver, and polymeric materials were produced.

Composites made of molybdenum fibers impregnated with silver were evaluated as static seals and showed good ability to seal air heated to 1000°F and retained pulsating pressures from zero to 500 psi.

The relationship between the fiber structure, impregnant, and final composite were studied.

Impregnation techniques and secondary work processes like machining are outlined in this report.

**Publication Review**

This report has been reviewed and is approved.

**For the Commander:**

S. T. Galzerano, Major, USAF  
Chief, Elastomers and Coatings Branch  
Non-Metallic Materials Division  
Materials Laboratory
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COMPOSITE INORGANIC RESILIENT SEAL MATERIALS

I. INTRODUCTION

This report covers work done under an extension to Contract No. AP 33(616)-57 and follows the summary technical report (WADC Technical Report 59-338 Part I) submitted May 1959. The material discussed in this report represents an uninterrupted continuation of the work which is directed toward the development of composite materials for static and dynamic seals.

Materials consisting of metallic fibers were felted and sintered, or brazed, to form porous metallic bodies or skeletons. Sintering is used to bond stainless steel fibers, and brazing to bond molybdenum fibers. The terms "bonded" and "unbonded" skeleton fibers are used throughout this project to refer to skeleton condition. "Bonded" indicates that the fibers are either sintered or brazed at their inter-crossing points. "Unbonded" refers to a condition where the fibers are jointed by mechanical interlocking achieved during the felting process.

Fiber skeletons are filled with softer metals or polymeric materials and are called "composites".

II. COMPOSITE MATERIAL DEVELOPMENT

This research program is being conducted to provide new inorganic resilient materials which will be used in improved high temperature seals. One possible solution to this problem would be the use of composite materials which combine the advantages of various constituent materials and avoid their drawbacks. The general approach toward development of these new materials is related to powder metallurgy, but it is new in that it involves the use of metal fibers. A porous metal skeleton of sintered, brazed, or merely felted fibers is the basic product of fiber metallurgy. Properties of the final skeleton or porous mat depend not only upon the metal selected but also upon the fiber dimensions, felt density, and sintering techniques. Such skeletons are not suitable for seal applications and must be impregnated completely with a second material to form two continuous phases. Since both phases are continuous, this configuration offers an optimum possibility of combining the properties of each material. Thus, a continuous rigid skeleton made of steel or molybdenum offers maximum support to a continuous, lubricating, conforming, impregnated phase such as silver, tin, magnesium, or elastomer. It should be pointed out, however, that magnesium is not a good lubricating-conforming impregnate.

The aim of this technique is to obtain a composite material which combines the conformability and lubricating characteristics of a soft metal or metal-non-metal mixture with the strength, creep resistance, recovery, and other properties of a stronger metal.

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During the first phase of this research program, material properties necessary for seals were derived from the standpoint of sealing mechanisms and environmental conditions involved. Theoretical studies were conducted to determine which materials and impregnants would meet these property requirements and make it possible to fabricate seals.

The properties which determine the acceptability of constituent materials for composite fabrication are:

1. **Melting Point**

   While the melting point is in itself an important property in an impregnant material, the metallurgical phenomena of re-crystallization, work hardening, and variation of strength with temperature are implied quantities that need to be evaluated. The skeleton material should not be dissolved by the impregnant at operating temperatures. If the impregnant is molten at the seal operating temperature, surface tension and impregnant viscosity become important.

2. **Yield or Shear Strength**

   The yield strength of the impregnant determines, in part, the clamping force which must be applied to the static seal. The shear strength is of primary concern in determining the influence of the impregnant on skeleton resilience. A combination of both of these strengths determines the composite conformability at the sealing surfaces. In general, both yield and shear strengths should be low in a good impregnant material. Materials used for dynamic seals must have low friction and wear properties. Impregnants having low interfacial shear strengths are preferred in dynamic applications.

3. **Wetability**

   The porosity in the composite material is a function of how well the impregnant wets the skeleton fibers. A material with good wetting characteristics will fill complex crevices in the skeleton completely, thus eliminating porosity.

4. **Stability of Properties**

   Ideally, the change in composite properties between room and 1000°F temperatures should be very small. Impregnant-skeleton systems which would involve phase changes, i.e., intermetallics or transformations, accompanied by a sharp variation in properties within this temperature range, must be avoided.
5. Minimal Alloying

The skeleton and impregnant should be selected for minimum mutual solubility at elevated temperatures. Higher solubility rates will change composite resiliency and other properties. Skeleton materials having a high modulus of elasticity are preferred for their resilience, and the impregnant must be metallurgically matched to the skeleton. There are many metallic materials which have a high modulus of elasticity, but only a few are available in quantity and are well enough known for sufficient engineering data to have been accumulated to permit their serious consideration as skeleton materials.

Cobalt, iron, and nickel are similar enough to permit the selection of a representative element. Iron in the form of stainless steel was selected to represent this group. Molybdenum is much easier to work with than tungsten and was, therefore, selected as representative of the higher modulus materials. Consideration will now be given to the impregnating materials for the two skeleton materials. All elements which have melting temperatures below 2000°F were considered and reactive and unstable materials such as radium, thallium, etc. were eliminated. The remaining elements are aluminum, barium, cadmium, copper, germanium, indium, tin, lead, magnesium, and silver. Neglecting, for the moment, their frictional and strength properties and considering only compatibility, a list of possible effects can be prepared (Table 1).

Intermetallic compounds are chemical compounds of two metals. They are usually stable and have fixed properties, but these properties differ greatly from the constituent metal. Aluminum and zinc both form intermetallic compounds with iron by partially dissolving them.

The metallic impregnants used in the experimental work were indium, tin, and silver. Indium has the most desirable friction, yield strength, and wetting properties. Properties of tin are not as favorable for impregnation purpose as indium. Tin was selected for these experiments because of its availability, the existence of a large body of engineering knowledge related to its use in bearings and other dynamic applications, and its low cost. Because they have low melting temperatures, neither tin nor indium is suitable as an impregnant for static seal materials intended to retain high pressures above their melting temperatures.

Thus far, the most promising impregnant metal for applications where high temperatures and pressures exist is silver. The best non-metallic impregnants are polymers.

III. COMPOSITE MATERIALS FOR SEALING PURPOSES

In order to evaluate composite materials and to determine their mechanical properties, suitable specimens of the required shape and size were made. Two different types of specimens were produced for these
## COMPATABILITY OF VARIOUS IMPREGNANTS WITH SKELETON MATERIALS

<table>
<thead>
<tr>
<th>Impregnant</th>
<th>Skeleton Iron</th>
<th>Molybdenum</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>SI</td>
<td>OK</td>
<td></td>
</tr>
<tr>
<td>Barium</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Cadmium</td>
<td>OK</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Copper</td>
<td>OK</td>
<td>OK</td>
<td>Work hardens, high yield strength</td>
</tr>
<tr>
<td>Germanium</td>
<td>I</td>
<td>I</td>
<td></td>
</tr>
<tr>
<td>Indium</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Lead</td>
<td>OK</td>
<td>S</td>
<td></td>
</tr>
<tr>
<td>Magnesium</td>
<td>OK</td>
<td>OK</td>
<td>Poisonous vapors, oxidizes, poor wear surfaces</td>
</tr>
<tr>
<td>Silver</td>
<td>OK</td>
<td>OK</td>
<td>Brittle, high yield strength</td>
</tr>
<tr>
<td>Tin</td>
<td>OK</td>
<td>OK</td>
<td></td>
</tr>
</tbody>
</table>

**KEY:**
- OK - Acceptable
- S - Dissolves skeleton
- I - Forms intermetallic compounds
- NA - Information not available

Purposes. Photographs shown in Fig. 1, 2, and 3 are specimens made for comparative functional evaluation. Specimens for property determination and conventional tests were made in accordance with ASTM standards and are not shown.

Figure 1 shows disk-like specimens which were used for comparative functional evaluation as static seals at room and elevated temperatures. These specimens were also employed in the determination of composite porosity. Specimens made of various constituents were evaluated and the results are presented in this report. The fixture and test set-up used are described in the Summary Report (WADC Technical Report 59-338) and are shown again in Appendix I of this report.

Specimens shown in Fig. 2 were made for comparative evaluation as dynamic seals on a reciprocating shaft. However, experiments and theoretical considerations indicated that a different geometrical configuration is required for this application. Although the test arrangement and fixture were described and illustrated in Quarterly Progress Report No. IV, they are shown again in Appendix I of this report.
Figure 3 illustrates the gaskets which were cut to size by General Electric of Cincinnati for their own applications. These were cut from 1/16-in. sheet made of 430 stainless steel sintered fibers having densities from 10 to 45 per cent. The gaskets are to be impregnated.

IV. MECHANICAL PROPERTIES

The mechanical and physical properties of fiber metal composites produced during this work period have been determined. Table 2 is a list of the properties of the constituents and composites.

One interesting aspect of the fiber composite materials is that, unlike the solid metallic materials, their properties can be changed to a considerable extent by varying the constituents and fabrication methods. It should be remembered, however, that the composite material does not acquire properties from its individual constituents in proportion to the amount of constituent present (Table 2).

As previously mentioned, the properties of principal interest for static seal applications are:

1. Yield strength or proportional limit
2. Hardness
3. Resilience
4. Melting point of impregnant
5. Oxidation or chemical resistance.

Yield strength can be increased easily by bonding or by increasing the density of the skeleton. But, unfortunately, if these methods are used to obtain higher strength, the hardness is increased and the resilience decreased. For example, unbounded Specimen No. 5 was deformed 0.05 inch/inch (5 per cent) with 1221 psi load and recovery was 94.6 per cent, while bonded Specimen No. 6 was deformed only 0.00133 inch/inch with 1477 psi load and recovery was 93.4 per cent. Resilience depends on many more variables and the method of obtaining a maximum value is not clearly discernable as yet. The following variables are known to influence resilience: fiber length and diameter, bond strength and interband distances, inter-constituent wetting, and the relation of gross sample size to sample internal structure. A good measure of the effect of these variables can be obtained by experiments.

For maximum validity, such experiments should closely simulate seal applications. This is especially important if more than one variable is under investigation.

The mechanical strength is higher for bonded skeleton fibers than for the corresponding unbonded skeleton. For example, a 27 per cent unbonded 430 stainless steel skeleton impregnated with tin has a yield strength of 4300 psi, while the same bonded skeleton exhibits a 16700-psi strength. The fiber composite is always weaker than the solid skeleton metal of which it is made. The strength is, however, adequate for seals. Materials for successful seal application are judged not only on the basis of their strength, but also on their resilience, conformability, and other mechanical properties. These properties can be obtained by choosing the proper constituents and fabrication methods.
Fig. 1 EXPERIMENTAL SPECIMENS FOR STATIC SEALING

Fig. 2 EXPERIMENTAL SPECIMENS FOR RECIPROCATING SHAFT SEALING

Fig. 3 GASKETS
## Table 2

### FIBER SKELETON AND COMPOSITE PROPERTIES

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Skeleton Material</th>
<th>Brinell Hardness Material</th>
<th>Density, g/cc</th>
<th>Modulus of Elasticity, 10^6 psi</th>
<th>Yield Strength, psi</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>Mo 160-185</td>
<td>16.2 5/8</td>
<td>0.004</td>
<td>18.48</td>
<td>50</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Mo 160-185</td>
<td>10.2 5/8</td>
<td>0.008 60</td>
<td>Unbonded</td>
<td>50</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>430 ss 145-185</td>
<td>7.70 3/4</td>
<td>0.0035 39</td>
<td>Bonded</td>
<td>29.0</td>
<td>40-55</td>
</tr>
<tr>
<td>7</td>
<td>Mo 160-185</td>
<td>14.2 3/4</td>
<td>0.004 39.4</td>
<td>Unbonded</td>
<td>50</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>430 ss 145-185</td>
<td>7.70 1/4</td>
<td>0.005 72.6</td>
<td>Bonded</td>
<td>29.0</td>
<td>40-55</td>
</tr>
<tr>
<td>9</td>
<td>430 ss 145-185</td>
<td>7.70 1/2</td>
<td>0.005 26.9</td>
<td>Bonded</td>
<td>29.0</td>
<td>40-55</td>
</tr>
</tbody>
</table>

### IMPREGNANT PROPERTIES

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Impregnant Material</th>
<th>Brinell Hardness of Impregnant</th>
<th>Density, g/cc</th>
<th>Modulus of Elasticity, 10^6 psi</th>
<th>Yield Strength, psi</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>Tin</td>
<td>5.3</td>
<td>7.3</td>
<td>6.5</td>
<td>1,250</td>
<td>0.2% offset</td>
</tr>
<tr>
<td>5</td>
<td>Indium</td>
<td>0.90</td>
<td>7.31</td>
<td>1.57</td>
<td>310</td>
<td>Compression Tension</td>
</tr>
<tr>
<td>6</td>
<td>Tin</td>
<td>5.86</td>
<td>7.30</td>
<td>6.0</td>
<td>1,500</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Silver</td>
<td>32.6</td>
<td>10.49</td>
<td>11</td>
<td>7,900</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Tin</td>
<td>5.3</td>
<td>7.30</td>
<td>6.0</td>
<td>1,250</td>
<td>0.2% offset</td>
</tr>
<tr>
<td>9</td>
<td>Magnesium</td>
<td>30.0</td>
<td>1.74</td>
<td>6.5</td>
<td>3,000</td>
<td>0.2% offset</td>
</tr>
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### COMPOSITE MATERIAL PROPERTIES

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Composite Density Number</th>
<th>Volume of Impregnant, % of Total Volume</th>
<th>Weight of Impregnant, % of Total Weight</th>
<th>Modulus of Elasticity in Compression, 10^6 psi</th>
<th>Yield Strength, psi</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>7.80</td>
<td>15.8</td>
<td>81.25</td>
<td>75.95</td>
<td>10</td>
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<tr>
<td>5</td>
<td>8.98</td>
<td>13</td>
<td>40.6</td>
<td>32.5</td>
<td>1.1</td>
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<tr>
<td>6</td>
<td>7.46</td>
<td>23.4</td>
<td>64.3</td>
<td>60.60</td>
<td>7.05</td>
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<tr>
<td>7</td>
<td>10.36</td>
<td>63.3</td>
<td>60.6</td>
<td>61.3</td>
<td>14.0</td>
</tr>
<tr>
<td>8</td>
<td>7.59</td>
<td>61.2</td>
<td>27.4</td>
<td>26.4</td>
<td>9.07</td>
</tr>
<tr>
<td>9</td>
<td>3.63</td>
<td>44.7</td>
<td>73.1</td>
<td>35.2</td>
<td>13.6</td>
</tr>
</tbody>
</table>

*0.2% offset

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Table 3 shows the deformation and recovery values obtained from the mechanical property test data. These tests were performed for the determination of modulus of elasticity in compression on various composite materials that were produced. Testing was conducted according to the standard ASTM procedures.

The deformation of these materials is smaller than would be expected for an elastomer at the same stress level. This deformation, however, is far greater than the deformation which would be experienced with a solid structural material subjected to the stresses shown.

Table 3
STRESS-STRAIN VALUES OBTAINED ON STANDARD ASTM TEST SPECIMENS

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Deformation inches/inch per cent</th>
<th>Stress, psi</th>
<th>Recovery, per cent</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.0500 5</td>
<td>1222</td>
<td>94.6</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0.0013 0.1</td>
<td>3477</td>
<td>83.4</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>0.0008 0.58</td>
<td>10,000</td>
<td>54.5</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>0.0006 0.05</td>
<td>10,165</td>
<td>85.7</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>0.02 2</td>
<td>6620</td>
<td>62.0</td>
<td></td>
</tr>
</tbody>
</table>

Plain Skeleton

<table>
<thead>
<tr>
<th>Density 20.2%</th>
<th>Deformation inches/inch 0.27</th>
<th>Stress, psi 382</th>
<th>Recovery, per cent 89</th>
<th>Remarks  Room temperature</th>
</tr>
</thead>
</table>

The recovery in all cases was less than complete, that is, less than 100 per cent, indicating that the compression exceeded the partial elastic limit of the material in all cases. This partial elastic limit may be explained as follows: At the start, few fiber columns are compressed due to fiber random deposition. As the compression is increased, the fibers reach and surpass the elastic limit, become plastically deformed, and do not recover.

Material No. 7 data shows two values for percentage of recovery. The lower recovery value was obtained on the initial testing of the sample. The higher value was obtained when the same sample was retested. The second deformation contributed to the impregnant metal shear strength. It restrains the movement of skeleton fibers during the first compression which starts in an unstressed condition, but does not allow them to return to a stress-free state. The stress in the skeleton may be understood if it is thought of as an elastic force which is exactly equal to the shear strength of the impregnant.
In the second load-unload cycle the recovery is much greater because the skeleton was compressed in a stressed condition. There was no length loss in stressing.

The incomplete recovery in the second test can be charged to plastic deformation or the breaking of bonds in the skeleton.

A. Skeleton Resilience

To evaluate the resilience of a non-impregnated fiber skeleton and to study this property at room and 1000°F temperatures, compression and recovery tests were performed. Specimens on which these tests were performed consisted of 430 stainless steel fibers, bonded, and having a 20.2 per cent density.

In room temperature tests this material exhibited spring-like properties as was expected. The spring constant K value was 43,000 lb/in., and recovered 89 per cent at 27 per cent deformation. Values at greater deformation could not be obtained due to buckling of the specimen during the compression. The fiber skeleton structure is inherently unstable as a column if it is not adequately reinforced or supported.

The elevated temperature test results were not consistent with the general thesis that steels become weaker with increasing temperature. The skeleton in this compression test exhibited a spring constant, K, value of 52,500 lb/in., and recovered 100 per cent at 0.27 per cent deformation. There are several possible explanations for this peculiar and contradictory behavior. The most obvious one concerns the oxide layer on the surface of the fibers which results from the sample's being heated in air. This oxide stiffens the fiber and increases the apparent skeleton strength. At the same time, oxidation would decrease the metallic portion of the fiber cross section. For a given deformation, the outer fiber stress in the oxidized sample is lower. Given a specific yield stress, the lower fiber stress would mean that a greater portion of the deformation of the oxidized fiber is elastic deformation. Since this elastic deformation is recoverable, a greater skeleton resilience could be expected. Several other explanations are possible and more experimental work is necessary to confirm these data before a theory can be developed.

These tests were performed using 0.500-inch outside diameter by 2.250-inch long specimens. This is a standard size specimen adapted by ASTM for modulus of elasticity determinations.

The diameter-to-length ratio was derived by ASTM to avoid test complications that arise when specimen geometry influences test results. The properties of specimens having these dimensions are the properties of the material under test. These properties can be applied to specific or peculiar geometries with relative ease and reasonable reliability. It is generally difficult to derive true material properties from data which are influenced by specimen geometry.

It was noted that the fiber skeleton specimen buckled during the test. If the skeleton is assumed to be composed of randomly-sized coil springs, the distribution of which is somewhat uneven, then compressive loading would tend
to deform the weak side and buckle the strong side in an uncontrollable manner. This is, however, a property of fiber skeleton material and as such will influence application. Therefore, these findings should be noted, not minimized or ignored. Buckling can be avoided if a short non-standard specimen is adopted. In this case, the test results would be meaningful only for that specific specimen size and such data would be almost impossible to apply in any other geometry. The impregnated skeletons avoid this buckling difficulty because the impregnated metal distributes the load and retards the local movements which differ from the general deformation.

The normal mechanical tests performed on rubber-like materials must, of necessity, be adjusted to sample size, hardness, area-to-thickness ratio, and slippage between the ends of the test sample and the compression loading fixture. The composite metallic materials have much higher resilience than the solid metals of which they are composed, but the differences between the properties of rubber and metallic composites amount to several orders of magnitude. Some of these differences are shown in Table 4 below.

Table 4

<table>
<thead>
<tr>
<th></th>
<th>Ultimate Strength Tension, psi</th>
<th>Maximum Elongation Tension, per cent</th>
<th>Stress at 0.01% Strain, psi</th>
<th>Recovery from 0.01% Strain, per cent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rubber</td>
<td>2500-3500</td>
<td>750-850</td>
<td>Less than 0.01</td>
<td>Essentially 100</td>
</tr>
<tr>
<td>Metallic Composite</td>
<td>50,000</td>
<td>24</td>
<td>15,800</td>
<td>87</td>
</tr>
</tbody>
</table>

These differences are such that it is possible to test composites by applying metal test procedures instead of the more complicated rubber test procedures. Actually, it is quite meaningless to apply rubber terms to metallic systems. The only really meaningful question is whether or not the respective materials do function as seals.

A new testing concept, different from that used for rubber or metal, must be developed in order to correlate the resilience properties of the composite metallics.

B. Machining

The techniques of sintering, coining, and bonding or sintering of fiber-metal bodies are fairly well established and various specimens for laboratory experiments and evaluation have been made. After these materials have passed the development stage and their superiority over conventional materials has been
well-established, these techniques can be modified and expanded for larger production of fiber metal component parts having various shapes and configurations. At the present time, however, the shapes which can be fabricated are limited. It is difficult, for example, to produce shapes with undercut, holes, and other configurations which need machining. However, some of the specimens needed for property study and functional evaluation had shapes which required some machining.

Difficulties were encountered when attempts were made to machine non-impregnated fiber metal bodies without special preparation, especially in the case of those which had low density and weaker bonds. Therefore, secondary working processes had to be developed. After a few attempts to impregnate the skeleton with a rigid material in order to improve machining qualities it was found that a salt mixture consisting of 44.5 per cent potassium nitrate (KNO₃) and 55.5 per cent sodium nitrite (NaNO₂) by weight is suitable for skeleton impregnation. The melting temperature of this mixture is approximately 300°F. The impregnation process is as follows:

The skeleton is placed in a glass container which closely approximates the fiber skeleton dimensions. This container is then lowered into a vibrating salt bath, the temperature of which is raised to 300°F. The skeleton is left in the vibrating bath until air bubbles cease to appear. Then, the container with the skeleton is removed and cooled to room temperature. The glass must be broken in order to remove the skeleton. Salt is water soluble and if the machining is not done immediately after the skeleton is removed from the glass container, the skeleton is dropped into molten wax in order to coat it and prevent moisture absorption from the air. After machining to the desired shape, the salt can be washed out with a cold water stream.

V. STATIC SEAL EVALUATION

One of the basic property requirements for gasket materials is that they will seal with low loading forces and will retain that seal at high temperatures. In the experimental evaluation, gasket loading is accomplished by tightening bolts between which the gasket is clamped. The magnitude of the load imposed on the gasket and the clamping force are directly proportional to the bolt torque. Bolt torque is used as a measure of gasket load because it is easily determined with a torque wrench.

Bolt torque measurements were made on various fiber metal composites in order to determine the value of these composites as static seals and to study their gas permeability and impregnant penetration. Figure 4 shows graphs plotted from the experimental data. Graphical illustrations of this experimental set-up are presented in Appendix II.

The measured bolt torque was increased at intervals and air pressure at the moment of leakage was recorded for each torque. This was done at room temperature. The curves in Fig. 4 represent the following materials:
No. 4 - 430 stainless steel fibers, bonded, 18.5 per cent density skeleton impregnated with tin

No. 5 - Molybdenum fibers, unbonded, 59 per cent density skeleton impregnated with indium

No. 6 - 430 stainless steel fibers, bonded, 40 per cent density skeleton impregnated with tin (HCl gas was used to improve wetting)

No. 7 - Molybdenum fibers, unbonded, 40 per cent skeleton density, impregnated with silver

No. 8 - 430 stainless steel fibers, bonded, 72 per cent skeleton density, impregnated with tin (HCl gas used to improve wetting)

No. 9 - 430 stainless steel fibers, bonded, 26.9 per cent skeleton density, impregnated with magnesium

Special Specimens a and b

Special - 430 stainless steel fibers, bonded, 47 per cent skeleton density, impregnated with an elastomeric polymer. This impregnant is an experimental inorganic polymer composed of crystalline trimeric or tetrmeric phosphonitrilic chloride and is deposited into the skeleton body by melting.

Fig. 4 Graph indicating comparative clamping forces on static seal flanges for maximum gas pressures that are retained
Tests were started at room temperature by forming a leak-tight seal in the manner already described. The whole assembly was then heated with an electrical heater to a specified equilibrium temperature and pressurized with compressed air to determine the pressures that the seal is able to retain. Finally, the assembly is cooled to room temperature and the loss in bolt torque is measured. The following observations were made during these experiments:

Material No. 4 Medium clamping forces were needed to seal air pressures up to 5000 psi at room temperature. A certain porosity was present in the sealant body indicating that the impregnant wettability was poor. A better than average seal was retained at temperatures below the melting point of the impregnant. However, when the temperature exceeded the impregnant melting point, the impregnant was extruded from the skeleton causing severe leakage.

Material No. 5 Despite the higher skeleton density, lower clamping forces were needed to retain air pressures up to 5000 psi at room temperature. This is due to the fact that skeleton fibers were unbonded and had a low impregnant modulus of elasticity. There was no porosity in the sealant body because indium has good wetting characteristics and complete impregnation of the skeleton was achieved. A good seal was retained at temperatures of approximately 100°F to 120°F below the indium melting point of 220°F. The bolt torque loss at this temperature was 20 per cent. When the temperature approached the melting point, the impregnant was forced out of the skeleton body causing leakage.

Material No. 6 This material was similar to Material No. 4 except that it had a higher skeleton density. The clamping forces to retain the same air pressures were higher and the material had a much lower porosity. A good seal was retained at temperatures approximately 200°F below the melting point of tin (450°F). The bolt torque loss at this temperature was 15 per cent. The impregnant was extruded from the skeleton body at temperatures approaching the melting point of tin.

Material No. 7 This material was made of molybdenum unbonded fibers and impregnated with silver. Impregnation was complete. There was no gas leakage through the material body when pressures of 5000 psi were applied at room and at 1000°F temperatures.

The sealing surfaces of the specimen for evaluations as a static seal were prepared by machining on a lathe and had concentric turning marks measured at approximately 50 rms. Average clamping forces were needed to retain gas pressures at room temperature (see Fig. 4). Pulsating gas pressures from 0 to 5000 psi were retained just as well at 1000°F temperature as at room temperature.

The fixture was cooled from 1000°F to room temperature and re-heated to 1000°F. After three such temperature cycling, pulsating pressures from 0 to 5000 psi were applied and again no leakage occurred. No bolt torque loss was found. A specimen made of pure silver was subjected to the same pressures and one temperature cycle had a 20 per cent bolt torque loss.
One specimen made of No. 7 material was subjected to the tests outlined above at higher temperatures. The fixture was heated from room temperature to 1200°F. Air pressure from 0 to 5000 psi was applied repeatedly (approximately 10 times) at this temperature, and no leak was detected.

Three such temperature cycles were made and pulsating pressures applied. The seal did not show any deterioration or leak. There was no bolt torque loss after cooling to room temperature.

Material No. 8 Attempts were made to evaluate a composite material consisting of 430 stainless steel bonded fibers impregnated with tin and having a density of 72.6 per cent. The intention was to determine what gas pressures can be retained with a denser skeleton at temperatures where the impregnant is in a molten state.

The specimen had spots where the impregnant did not penetrate and when the bolts were tightened, pores inside the composite remained. Gas pressures over 200 psi leaked and the experiment had to be discontinued.

Material No. 9 Medium flange pressures were required to retain 5000 psi gas pressures. When the fixture was heated to approximately 1010°F, gas pressures were retained after cycling from zero to 5100 psi. After cooling to room temperature and repressurizing, gas leakage occurred at 3000 psi. More leakage was found after several pressure cycles at room temperature. Bolt torque loss amounted to 69 per cent.

Special Specimen Two disk-like specimens were impregnated with an experimental polymer (polyphosphonitrillic chloride) and subjected to the static seal evaluation. Because the first impregnation attempts did not produce identical composite materials they were denoted as specimens "a" and "b". Material a, sealed high pressures with the lowest clamping forces. Material b required somewhat higher clamping forces. Some porosity was present in both materials, but was easily eliminated with low clamping forces. Gas pressures up to 5000 psi were retained at room temperature. The temperature was then raised to approximately 820°F. After a two-hour exposure to this temperature, the pressure cycling from zero to 5000 psi was applied. Slight leakage was noticed after 15 cycles and leakage gradually increased with additional cycles. No bolt torque loss was found when the fixture was cooled to room temperature.

The evaluation data indicate that impregnants having a melting point slightly above the seal operating temperatures are acceptable providing that they possess the other required properties such as good wetting, non-solubility, etc. Any skeleton material which has unlimited solubility in the impregnant material at operating temperature would soon lose its identity. In general, the properties of a material change with increasing temperature. These changes include thermal expansion, solidification shrinkage, and chemical solubility or oxidation. Fabrication difficulties are introduced by these changes. An acceptable compromise between the required properties and the specific seal application need to be found.

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Inter-constituent wetting, whether it is by limited solubility or by close mechanical association, appears necessary to resist impregnant ex- traction and to eliminate porosity. A method for suppressing this extrusion without loss of sealing capacity may be to use an alloy which is slushy (two-phase) at operating temperature. Such an alloy, unlike tin, would be jelly-like and could resist greater pressure before being forced out. Another possibility is the use of a third constituent in powder form which would be wetted, but not dissolved, by the impregnant metal. The impregnant in turn should partially dissolve the skeleton metal so that it would not impair the skeleton's resilience but would encapsulate the metal. Under conditions outlined above could be provided, it is possible that the skeleton structure would retain the powder constituent and the powder would retain the liquid metal by surface tension forces.

The skeleton materials, on the other hand, must possess high strength and temperature resistance. In this respect, molybdenum with its modulus of elasticity and high strength, is one of the most promising skeleton materials.

VI. DYNAMIC SEAL EVALUATION

Attempts were made to evaluate the most promising composite materials as sealants in a reciprocating shaft arrangement to seal MLO 57-461 high temperature hydraulic fluid. The test arrangement used for these experiments is shown in Appendix II. Seal dimensions and configuration are shown and discussed in Appendix I. Two different composite materials were made for this evaluation. One was silver impregnated composite designated as Material No. 7; the second material was impregnated with an inorganic polymer designated as Special Specimen. The other materials having low melting temperature impregnants were not used because the experiments with static seals indicated that they are extruded at impregnant melting temperatures and high pressures.

The experimental set-up has a 1/4-inch diameter reciprocating shaft and a stroke of up to 4 inches is obtainable. By tightening the nut, the sealant is pressurized around the shaft in a packing gland arrangement. Pulsating pressures are controlled manually. The purpose of this simple arrangement is to make comparative material evaluation, i.e., to determine which material performs better during a set time interval and temperature. The ones showing the most promise can be evaluated as seals on a full-scale experimental set-up.

A composite material sealant made of molybdenum fibers and impregnated with silver were placed in a fixture, pressurized, and a regular reciprocating shaft movement was employed. Only scattered evaluation data could be obtained because the sealant's shape and configuration were not suitable for low resilience material.

Additional specimens of this material were produced. Instead of experimenting further with our miniaturized test system, arrangements were made with Republic Aviation to evaluate them on their full-scale test set-up. Material samples have been mailed to them. Republic Aviation Corporation, Farmingdale, New York (USAF Contract AF 33(610)-6411) has undertaken, in part, the development work and testing of high temperature seals.
Sealants of the same size and shape were made of material consisting of 430 stainless steel fibers impregnated with an experimental inorganic rubbery polymer. This material is much more resilient than impregnated silver, and a better seal of pressurized liquid was obtained without change in configuration. Experimental evaluation work had to be discontinued because of specimen availability. Plans are being made to produce additional material of this type.

VII. IMPREGNATION AND APPARATUS

Fiber skeletons, either bonded or unbonded, were impregnated with filler materials. Impregnation methods and apparatus have undergone certain changes and innovations since the beginning of the project. Graphical illustrations of the set-ups used for the latest specimen impregnation are shown in Fig. 5, 6, and 7.

This apparatus is an improvement and refinement over the sample fabrication techniques. However, it is not yet considered to have attained its final form. This fact precludes the construction of a permanent fabrication apparatus, however, it is not envisioned that the ultimate conversion of laboratory techniques to production will involve any particular difficulties.

Illustration of the tin impregnation set-up is shown in Fig. 5. The skeleton and the tin are placed in a container as shown in the graphical illustration. Air is evacuated, HCl gas injected, and the temperature raised to 750°F, at which tin boils vigorously. After sufficient boiling to clear the molten metal of SnCl₂, the impregnation takes place almost instantaneously upon the admission of argon pressure.

Figure 6 shows the silver impregnation set-up. First, the skeleton and silver are placed in a container as shown and the air is evacuated. The temperature is raised to 2000°F and the silver melts. When the silver is all molten, argon is admitted to the container and the liquid metal is forced into the skeleton pores.

The graphical illustration shown in Fig. 7 represents the magnesium impregnation set-up. In a manner similar to that used in the two impregnation processes described above, constituents are placed in the container and the temperature is raised to 1500°F where the impregnation takes place in an argon gas atmosphere. Magnesium reacts with steel and is self-fluxing. The process, however, does produce gas inclusions inside the skeleton body, thus introducing porosity in the composite material.

A. Skeleton Impregnation with Polymeric Materials

Polymeric materials and, in particular, the so-called inorganic polymers, exhibit resistance to temperatures up to 100°F. Such polymers are being produced, but the reports indicate that their very poor mechanical properties at 100°F would make them unsuitable for any application. These materials may be useful as fillers or impregnants for metallic fiber skeletons because of low stresses necessary to produce the required deformations.
Fig. 5 GRAPHICAL ILLUSTRATION OF TIN IMPREGNATION SET-UP
Fig. 6 GRAPHICAL ILLUSTRATION OF SILVER IMPREGNATION SET-UP
Fig. 7 GRAPHICAL ILLUSTRATION OF MAGNESIUM IMPREGNATION SET-UP
Composite materials already produced of steel fibers and im-
pregnated with softer metals like silver or tin had higher resilience than the
solid metals of which they are composed, but the stresses for the same a-
mount of strain are much higher than would be needed for elastomer im-
pregnant. Since high-temperature applications are contemplated, only high-
temperature-resistant polymers can be considered. It is expected that com-
posites with these impregnants may be useful at temperatures up to the point
of polymer decomposition.

Impregnation difficulties were experienced with metallic impregnants
mainly because of inadequate wetting and lack of a permanent experimental
set-up. One main barrier to proper impregnation of polymeric material into
the skeleton body is the solid state in which they are available. For example,
attempts were made to impregnate Teflon lattices or solutions into skeleton
body but the porosity could not be eliminated even after repeated impregnation
where the solvent was removed between impregnations.

Very good impregnation results were obtained with an inorganic
polymer (phosphonitrillic chloride) known as "inorganic rubber". This im-
pregnant was selected, not because of its particular thermal stability, but
because of its easy preparation and because it can be applied by a melt-im-
pregnation technique. In this case, there is no void problem because no
solvent is used.

Phosphonitrillic chloride rubber is sensitive to moisture and the
exposed seal surfaces are subjected to hydrolysis in the presence of moisture.
It will be necessary to stabilize that part of the seal by subjecting its surface
to a chemical reaction which would remove the chlorine atoms from the
surface layer and replace them with stable groups.

Some references indicate that this elastomer is expected to retain
its properties to 600°C, but is reported to be converted to a hard material
of unknown composition when the temperature is raised to red heat.

The first attempts to impregnate skeletons made of metal fibers
with this elastomer were performed as follows:

The skeletons were placed in a pipe nipple closed at the bottom with
a cap. Mixed crystals of trimer (\(\text{FNCI}_2\)) and tetramer (\(\text{FNCI}_3\)) were
produced and the skeleton surface covered with them. The crystals were
melted at about 250°F, then gravity caused the melted crystals to penetrate
the skeleton body. Next, the nipple was closed with a cap and the assembly
heated overnight at about 575°F to produce the rubbery polymer. The im-
pregnation could be improved further by the adaptation of vacuum techniques.
This approach and the resultant composite material show promise. However,
the problem of improving the thermal stability remains.

B. Ceramic and Other Impregnants

The skeleton material has spring-like resilience with a spring con-
stant which is a function of void space sizes, density, and fiber dimensions.
Moreover, the resilience of the fiber skeleton is exhibited in all directions
while the spring is strictly unidirectional.
The material resilience property is highly desirable in seal applications, but the skeleton as such cannot be used unless its porosity is eliminated by impregnation with other materials. The impregnated material should possess properties like softness, conformability, and be in such a form that will be possible to squeeze it into the skeleton's void spaces. Furthermore, the skeleton's resilience should remain after the void spaces are filled with impregnant.

Functional evaluation tests indicated that composite materials impregnated with tin, indium, or other low-melting temperature impregnants are not suitable for seal applications where pressures are to be retained at ambient temperature approaching the impregnant melting point because the impregnant is extruded from the skeleton body. To prevent the extrusion of impregnant, materials should not melt at operating temperature or they should be in a slushy, jelly-like condition in order to resist extrusion from the skeleton body.

A study of ways and means to impregnate fiber metal skeletons with refractory materials was conducted. Attempts to impregnate fiber skeletons with colloidal materials, ceramic solutions, and sprayed coatings were unsuccessful mainly because the impregnation apparatus and methods were inadequate.

An obvious method of impregnation is to soak the fiber metal skeleton in a solution or dispersion of the refractory material. To displace trapped air and aid penetration, soaking should be done under vacuum. The following procedure was used in the impregnation attempts discussed here: Specimens were placed in a container with impregnating solution, enclosed, and connected to a vacuum desiccator. Bubbles were apparent which indicated that the skeleton void spaces were being filled with solution replacing air. The bubbling subsided within 2 to 3 minutes. A vacuum of about 20 inches of mercury was applied and the skeleton was soaked for 15 minutes. The specimen was removed and oven-dried at 212°F for approximately 10 minutes. The vacuum soaking was then repeated for an additional 15 minutes. Finally, the specimen was oven-dried at 212°F for 30 minutes.

This impregnation method is suitable for liquids. However, with slurries or solution containing particles of appreciable size the skeleton is not penetrated and other means of forcing these solutions into the skeleton body must be employed. Preparations are under way to make a set-up where vibrational forces will be used to squeeze these materials into the skeleton.

The following colloidal materials and solutions were used as impregnants:

1. Nalcoag Grade 35. Contains 35 per cent silica, pH about 7.5 in water. The supplier is National Aluminate Corporation, Chicago.

2. Aqueous Colloidal Zirconia. Contains 25 per cent zirconia in water, pH about 2. The supplier is duPont deNemours, Experimental product.

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5. One specimen was coated in "Solution Ceramic", an aqueous solution of chromium nitrate, 10 per cent by weight, Cr(NO₃)₃ . 9 H₂O. This specimen was heated to 350°F and sprayed with this solution.

6. Teflon 30 dispersion containing 60 per cent solids. The supplier is duPont de Nemours. The specimen was first soaked in this solution. After 15 minutes, of soaking and then drying, the specimen was coated by spraying over a zircon-aluminum phosphate slurry. The slurry coat hardens at room temperature and becomes rigid and brittle upon heating. To expel water and stabilize aluminum phosphate, bond specimens were baked at 600°F for about 20 minutes. Teflon is not necessarily the ultimate in fluorocarbon polymers, but it was used for these experiments because of its availability. The intention was to fill skeleton voids with resilient material and yet have the specimen somewhat resistant to heat. The exterior ceramic coating was used to form a barrier against leakage.

As already mentioned, none of the impregnants used densified the skeleton body to the extent that pressurized gas leakage could be prevented. Some of the materials listed above will be used again as impregnants when the new impregnation set-up is assembled.

VIII. CONCLUSIONS

Composite materials can be produced with different and unusual properties by varying combinations of metallic fiber sizes, skeleton densities, and bonding, and by choosing the soft metallic or polymeric impregnants.

It has been demonstrated that materials consisting of fibers made of high melting temperature metals like molybdenum and impregnated with soft metal like silver have suitable properties for high temperature static seal applications.

Stainless steel fibers impregnated with polymers exhibited high resilience, and the work has indicated that the upper temperature limit of high temperature polymers can possibly be extended by using them in composite materials.
Experimental comparative evaluation was made to determine which materials are best suited for seal applications. The information obtained has not yet been resolved into workable design formulae and parameters, either because the promising materials have not been produced in sufficient quantity for further evaluation, or because those produced did not show much promise during preliminary evaluation.

Progress has been made toward the goals set for this project, but appreciable further work will be needed to select optimum constituents and impregnant combinations before a composite material demonstrating the full potentialities of this approach can be developed.

IX. RECOMMENDATIONS FOR FUTURE WORK

As a result of the basic work done on this subject, both in composite material fabrication and evaluation as seals, it follows that for a better understanding of the factors like resilience, influence of impregnants on composite properties, etc., further investigation such as that outlined below is needed.

1. Explore the availability of high temperature resistant polymers and use them as impregnants.

2. Produce more of various composite specimens to enable the study of seal shapes and friction on dynamic seals.

3. Investigate the usefulness of composites impregnated with indium for static and dynamic sealing of cryogenic liquids.


APPENDIX I

SEAL GEOMETRICAL CONFIGURATION
The initial work phase primarily involved development of fabrication techniques that would enable preparation of reproducible composite material specimens for the evaluation program. A number of different specimens were produced and the effects of certain fabrication techniques and constituents of composite properties were studied and evaluated in the light of intended application.

As already described, material evaluations as a static seal were made. No definite design parameters were established since the emphasis was on material development; however, comparative results on those materials which show promise for use as static seals were obtained. Evaluation of material as a dynamic seal (for example, as a packing to seal the reciprocating shaft) is more complicated. To obtain similar comparative results as was done with static seals, it is necessary to consider geometrical shape and seal configuration as well as properties such as low friction, temperature resistance, and strength.

The shape of the packing seal which was used for various refractory material screening tests and also for preliminary comparative evaluation of composite materials is shown in Fig. 8. The configuration was selected because of its simplicity and also because it served the purposes as outlined above.

As work progresses further and more specimens are produced for experiments there is a need to determine the seal shape and configuration for actual sealing purposes. Some of the theoretical considerations and simple analyses related to the application of composite material to the sealing of reciprocating shafts are given below.

![Fig. 8 EXPERIMENTAL PACKING](image)
Fiber metal composites are assumed to be isotropic materials, i.e., they have the same strength and elastic properties in all directions. Such materials exhibit a spring-like behavior when stressed in tension or compression. They are able to store or absorb energy, then to release it whenever needed in a particular application. Energy storage is accomplished by applying the load in one direction causing a dimensional decrease or material displacement in that direction and an expansion sideways. This expansion is utilized in seal applications to keep sealing surfaces under constant pressure and also to compensate for worn out material without appreciable pressure reduction between the surfaces.

A theoretical analysis of the experimental packing (shown in Fig. 8) is presented in order to establish fundamental design criteria and to draw attention to the shortcomings of the present configuration which is used in the comparative evaluation. The seal is compressed with load applied centrally at one end in such a manner as to avoid non-uniform stress distribution at any section of the seal. Using known stress and strain relationships, the following formulae are obtained:

Let the applied load be \( P \) lb

Cross-sectional area under load \( A \) in.\(^2\)

Assuming that the cross-section is uniform through the whole length and the stress is:

\[ S = \frac{P}{A} \text{ psi} \]  

(1)

If the section is not uniform and changes gradually, the stress at any section can be found by dividing the load by the area of that section; the total longitudinal deformation over a length \( n \) is given by:

\[ dS = \frac{P}{AE} \text{ dn} \]  

(2)

If the stress exceeds the proportional limit in the tapered seal section, the formulae above still hold, but the deformation and the work done in producing it can be determined only from experimental data.

The seal shortens under compression and the unit longitudinal strain \( y \) will be:

\[ y = \frac{S}{E} \]  

(3)

where \( E \) is modulus of elasticity.

The total longitudinal strain in the length \( m \) is \( e \):

\[ e = m \cdot y \text{ in.} \]  

(4)

or, substituting the values from Eq (1) and (2) into Eq (3),

\[ e = \frac{P}{AE} \text{ in.} \]  

(5)
The seal expands under compression. The unit lateral strain is the same in all transverse directions:

$$x = vy$$  \hspace{1cm} (6)$$

where $v$ is Poisson's ratio,

$$v = \frac{3K - E}{6K}$$  \hspace{1cm} (7)$$

where $K$ is Bulk Modulus of Elasticity.

Both longitudinal and lateral strains are proportional to the applied load provided that the stresses are uniform. Some materials exhibit equal stress distribution on uniform sections. Assuming that the stress is uniform and the diameter $D$ is kept constant by the rigid gland housing, the total lateral strain $z$ will be proportional to the lateral dimension $d$. This is expressed by

$$z = dx = dy =tv\frac{S}{E}$$  \hspace{1cm} (8)$$

Composite material when compressed, remains constant in volume under all ordinary loads. Thus the seal increases in the cross-section area as it is compressed and, in order to maintain a constant volume, the inside diameter should decrease. This decrease in diameter is accomplished by strain energy and takes the form of radial forces pressing the seal material around the shaft, thus closing the leak passages.

To obtain the initial seal and to preserve it at elevated temperatures while the shaft is reciprocating, these radial forces must be maintained at all times. A reserve of such forces is also needed to compensate for wear out or natural removal of seal material during prolonged operation. The material should be resilient and isotropic, so that vibrations and non-axial shaft loadings will be absorbed without opening leak passages.

The present seal geometry used for preliminary material evaluation does not provide the reserve radial forces needed to compensate for material wear and is not expected to maintain a seal for prolonged operation.

Changes of seal material geometrical form and dimensions are produced by stress. Higher stress will produce larger strain or displacement. Therefore, the seal geometry should be arranged so that the external force will produce stresses distributed in a way which will give the most effective sealing.

The stress distribution in seals (Fig. 8) may be assumed to be uniform where the cross-section area is uniform. The tapered end of this seal will, however, have gradually increasing stresses as the cross-section area becomes smaller. Thus, more actual reduction in inside diameter and more sealing will be done at this end.
To make an analytical or experimental stress-strain analysis of a complicated geometrical seal configuration would be difficult and unreliable for practical applications. However, certain design parameters can be established by combining experimental data with material properties, seal volume and material ability to store or absorb mechanical energy.

The strain energy stored up in stressed material is equal to the work done by the external forces in producing the stress and is recoverable if the magnitude of imposed stresses is below the yield strength.

The expression of strain energy per unit volume is:

\[ W = \frac{1}{2} \frac{S^2}{E} \frac{\text{in.} \cdot \text{lb}}{\text{in.}^2} \]  

(9)

Total strain energy is:

\[ W_T = \frac{1}{2} \frac{S^2}{E} V \text{ in.} \cdot \text{lb} \]  

(10)

where \( V \) is seal volume.

Also

\[ W_T = \frac{1}{2} P \epsilon \text{ in.} \cdot \text{lb} \]  

(11)

where \( P \) is applied load in pounds, and \( \epsilon \) is the total longitudinal strain in inches. Although composite materials do not follow Hooke's law precisely, these formulas can be used to express sealing efficiency in terms of seal volume and apparent energy versus reciprocating shaft diameter and seal configuration.

Using the experimental data from Table 3 and some of the stress-strain formulas, numerical values can be obtained and compared.

Now consider composite material samples No. 5 and 7 (Table 3) and seal dimensions which were used for the experiments (Fig. 9).

![Dimensions of Experimental Seal](image)

**Fig. 9** DIMENSIONS OF EXPERIMENTAL SEAL
Load is applied on seal area $A$:

$$ A = \frac{P}{4} (d^2 - d'^2) = 0.785 \left(0.625^2 - 0.25^2\right) = 0.258 \text{ in.}^2 $$

Total longitudinal strain is:

No. 5 $\varepsilon = 0.05 \times 0.375 = 0.01875 \text{ in.}$
No. 7 $\varepsilon = 0.000555 \times 0.375 = 0.00021 \text{ in.}$

(Note: stress concentration in the seals tapered end was not taken into account.)

Assuming Poisson's ratio $\nu = 0.3$, the total lateral strain $\varepsilon$ is, from Eq (8),

No. 5 $\varepsilon = \nu \times 0.05 = 0.25 \times 0.3 \times 0.05 = 0.00375 \text{ in.}$ (equals approximately 94 microns)
No. 7 $\varepsilon = \nu \times 0.000555 = 0.25 \times 0.3 \times 0.000555 = 0.00004 \text{ in.}$ (equals approximately 1 micron)

The forces required to compress sealant to its proportional limit are:

No. 5 $P = S \times A = 1222 \times 0.258 = 315 \text{ lb}$
No. 7 $P = S \times A = 10165 \times 0.258 = 2623 \text{ lb}$

The dimension of displaced material, or lateral strain, is of prime importance in determining the amount of pressure which will be obtained at the sliding shaft and material sealing surfaces, how well the worn material is replenished after prolonged operation, and how well compensation will be made for assembly misalignments and vibrations. The lateral strain and compression force are a measure of material resilience properties. These two composite materials (No. 5 and No. 7, Table 3) were selected to demonstrate the importance of resilience in dynamic seal.

Material No. 5 is impregnated with indium. Material No. 7 is impregnated with silver. Indium has a low modulus of elasticity and was deformed 94 microns with relatively low forces. On the other hand, the silver impregnated specimen, which has a much higher modulus of elasticity, was deformed only 1 micron by a force 8 times larger than that applied to the indium.

The deformation of 1 micron means that the assembly alignment and sealing surface roughness have to be within this dimension. Such a tolerance is almost impossible to achieve and a seal which was made from silver impregnated composite material did not perform well due to low resilience.
properties. In order to utilize the high temperature properties of this material, the seal geometrical configuration will be changed in the future by incorporating smaller cross-sectional areas which will exhibit higher deformation with lower compression forces.

Composite material resilience properties may be expressed as the amount of energy that can be stored. Taking the two materials again and using Eq (11) we have:

\[
\text{No. 5 } W = \frac{1}{2} P e = \frac{1}{2} 315 \times 0.01875 = 3 \text{ in.-lb}
\]

\[
\text{No. 7 } W = \frac{1}{2} P e = \frac{1}{2} 2623 \times 0.00021 = 0.27 \text{ in.-lb}
\]

This demonstrates that material having higher resilience is able to store more energy and, therefore, is better suited for seal applications.

Numerical value of the stored energy, if experimentally derived for particular seal configuration, can be used as one of the composite quality parameters in future material selection. The ability to store energy may be used as a criterion in the evaluation of a material’s suitability for sealing purposes.
APPENDIX II

APPARATUS AND ARRANGEMENT FOR
STATIC AND DYNAMIC SEAL EVALUATION
Fig. 10  ARRANGEMENT AND FIXTURE FOR STATIC SEAL EVALUATION
Fig. 11 FIXTURE AND SET-UP FOR RECIPROCATING SHAFT SEAL EVALUATION