ANTISEIZE AND THREAD SEALING COMPOUND FOR AIRCRAFT OXYGEN SYSTEMS

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L. W. Elston

Engineering Experiment Station
Georgia Institute of Technology

DJV

Contract No. AF 33(616)-6090

AEROSPACE MEDICAL LABORATORY
WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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FOREWORD

This report covers research on antiseize and thread sealing compounds conducted by the Engineering Experiment Station of Georgia Institute of Technology, with Mr. W. H. Burrows as principal investigator. The objective of this research was the development of a compound, incorporating solid and liquid lubricants, capable of sealing threaded pipe joints against pressures of oxygen up to 2000 psig and operative at temperatures from -65° to +600° F.

This report was prepared for the Aircraft Equipment Section, Engineering and Development Branch, Aerospace Medical Laboratory, Wright Air Development Center, under Contract No. AF 33(616)-6090, Project No. A-414. Work began October 1, 1958 and was completed May 31, 1959.

The authors are indebted to the staff of the Georgia Tech Research Institute for their help in handling the many administrative details of this project, to the staff of the Engineering Experiment Station for assistance in the preparation of reports, and to the staff of the Industrial Products Branch for constant assistance in the conduct of the research. Contracting agency for this project was the Georgia Tech Research Institute, Research Building, Georgia Institute of Technology, Atlanta 13, Georgia.

ABSTRACT

A study has been conducted to formulate and develop an antiseize and sealing compound from commercially available materials for use in aircraft oxygen systems in the -65° to 600° F temperature range. No formulation was stable under 2000 psig oxygen at 600° F for extended periods, but one compound exhibited satisfactory antiseize and sealing properties despite decomposition of its liquid phase. Compounds have been formulated by blending finely divided solids as antioxidants, thickeners, fillers, and inhibitors in the several commercially available silicone and siloxane fluids stable at temperatures near 500° F. Suitable thickeners, fillers, and solid lubricants are available, but further work is necessary to improve the stability of base fluids in the environment specified.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

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I. INTRODUCTION

The purpose of this first study is the development of an antiseize and sealing thread compound for aircraft oxygen systems. The requirements for this compound as defined in Specification MIL-T-5543B(ASG)[1] are that it be a stable, homogeneous material of greaselike consistency, that it be suitable for use from -65° to 600° F under 2000 psig oxygen, and that it be physiologically harmless in the environment. The scope of the study was formulation from existing materials rather than synthesis of new temperature-resistant liquids and solids.

A literature survey of materials suitable over the specified temperature range revealed no liquid stable under oxygen at 600° F and only a very limited number of fluids serviceable in the 500° F area.[2,3] Inquiries directed to manufacturers of high temperature lubricants brought forth even less encouraging suggestions. Experimental effort was directed toward blending various solid lubricants, antioxidants, and inhibitors with several of the most thermally stable liquids available to form a thread compound which would perform for as long a time as possible at temperatures in excess of the decomposition temperature of the fluid alone.

Manuscript released by the authors 1 October 1959 for publication as a WADC Technical Report.

II. EXPERIMENTAL WORK

A. Screening of Base Fluids

A survey of technical literature, industrial bulletins, and documents from the Armed Services Technical Information Agency indicated several possible base fluids.[2,3,4,5] As lubricity in the formulation could readily be imparted through the solid phase, shear strength was considered less important than thermal stability and low solidification points. We could not find any reference to a fluid that was designed for operation in open systems or in systems containing oxygen under high pressures at elevated temperatures

Initial screening of the base fluids was carried out using 5-gram samples of each liquid in open porcelain crucibles. The samples were placed in a muffle furnace and maintained at 600° F for 24 hours. Periodic observations of physical characteristics are summarized in Table I.

Performance in these trials indicated that Dow Corning Silicone fluid 710 (methyl phenyl silicone) shows the least deterioration in the presence of air at 600° F. This observation is in agreement with observations made by other investigators of extreme high temperature lubricants.[6,7] Polyphenyl ethers were considered and rejected because of their high melting points and low oxidative stability.[6,7]

TABLE I

OPEN CRUCIBLE SCREENING OF BASE FLUIDS AT 600° F

Material	Period of Exposure			
	10 Minutes	1 Hour	6 Hours	24 Hours
Dow Corning QF-6-7009	Fuming, charred odor	Fuming, charred odor	Fuming, charred odor, some thickening	Very little, slightly charred, residue
GE 81644	Fuming, charred odor	Fuming, charred odor	Fuming, charred odor, some thickening	Gel, slight fuming
GE Versilube F-50	Fuming charred odor	Fuming, charred odor	Fuming, charred odor	Very little, slightly charred, residue
GE 81705	Fuming, charred odor	Fuming, charred odor	Fuming, charred odor	Gel, very slight fuming
Dow Corning QF	Fuming, charred odor	Fuming, charred odor	Fuming, charred odor, some drying	Very slight residue
Dow Corning 200	Fuming, charred odor	Fuming, charred odor	Thickening, fuming, charred odor	Slight charred residue
Dow Corning 710	Slight fuming, odorless	Slight fuming, odorless	Slight fuming, odorless	Slightly thickened yellow liquid, slight fuming
Dow Corning 710R	Fuming, sharp, acrid fumes	Acrid fumes, test discontinued	---	---

B. Screening of Solid Materials

1. Thickeners and Fillers

As the silicone base fluids possess only limited shear strength and oxidative stability, it is necessary that a large portion of the compound's lubricity and oxidative stability be derived from the solid materials incorporated as thickeners and fillers. These solids must possess sufficiently high surface area to adsorb and hold the base fluid to form an homogeneous, grease-like substance which neither thins nor separates on standing. They must also impart sufficient body to the sealant to resist displacement under extreme pressure and temperature. As no single material possesses all of these properties, it was necessary to include several solids in the formulation in such proportions as to produce maximum benefit without exceeding for each material some limit beyond which the over-all effectiveness of the sealant is decreased. No literature was found describing lubricants to function in an environment of 2000 psig oxygen at 600° F and with low tolerance for toxicity of either the lubricant or its decomposition products. The choice of the solid materials and their optimum levels was made by trial and error after a study of the literature[5,7] with respect to each component. The sealants formulated with each of these solids and diphenyldidodecyl-silane[8] (Dow Corning fluid QF-6-7009) are listed in Table II. These materials exhibited such decomposition in open-crucible tests that bomb testing under 2000 psig oxygen was omitted.

A more extensive selection of solids[9] was screened using methyl phenyl silicone (Dow Corning 710 fluid) as a base liquid. As the upper limit of thermal stability for this fluid approaches the 600° F operating temperature, sought for the sealant formulation, smaller contributions are required from each of the solid materials included. Generally, each of the solids makes a single principal contribution as a thickener, filler, lubricant, or antioxidant, although a material included for one purpose might produce additional benefits in another area (e.g., some increase in lubricity is obtained by incorporating talc as a filler). These trials are summarized in Table III.

2. Solid Lubricants

As nearly all of the samples tested exhibited some drying on prolonged exposure to air at 600° F, it seemed desirable to substitute a solid lubricant for some or all of the filler and thickener in the sealant. This procedure, if practicable, would produce the necessary antiseize properties even if the base fluid were partially destroyed. Formulations employing two grades of molybdenum disulfide and three base fluids were examined. High levels of molybdenum disulfide produced a sharp increase in the decomposition rate of the base fluid. Decreasing the level of molybdenum disulfide permitted its use without complete hardening of the sealant in 24 hours' exposure to air at 600° F. These trials are summarized in Table IV.

3. Inhibitors and Antioxidants

Failure of the liquid phase was the most obvious common characteristic of all the unsatisfactory formulations. This failure could be attributed to vaporization, oxidative cleavage followed by vaporization, polymerization of the fluid, or polymerization of oxidation products of the fluid. It seemed probable

TABLE II

OPEN CRUCIBLE SCREENING OF FILLERS IN DOW CORNING QF-6-7009 FLUID

Sample No.	Composition	Compounding Method	Open Crucible 600° F, 24 Hours	Open Crucible 500° F, 24 Hours
1	27 g DC QF-6-7009 fluid 25 g Dixon's Microfyne graphite	Oil added gradually over 24 hours on ball mill	Dry, slightly charred powder	Hardened, no charring
2	24.5 g DC QF-6-7009 fluid 25 g No. 1 Lo Micron talc	Oil added gradually over 24 hours on ball mill	Dry, slightly charred powder	Soft, no charring (hardened in 72 hours)
3	33 g DC QF-6-7009 fluid 25 g Micro-Cel	Oil added gradually over 24 hours on ball mill	Charred cake	Not attempted
4	28 g DC QF-6-7009 fluid 6.25 g graphite 18.75 g No. 1 talc	Oil added periodically over 24 hours on ball mill	Dry, slightly charred powder	Hardened, no charring
5	10 g DC QF-6-7009 fluid 0.1 g aluminum powder MD3100 10 g graphite	Mortar and pestle, 30 minutes	Not attempted	Charred cake
6	10 g DC QF-6-7009 fluid 0.2 g aluminum powder MD3100	Mortar and pestle, 30 minutes	Not attempted	Cake, no charring
7	10 g DC QF-6-7009 fluid 0.5 g aluminum powder MD3100 10 g graphite	Mortar and pestle, 30 minutes	Not attempted	Charred cake
8	10 g DC QF-6-7009 fluid 1 g aluminum powder MD3100 10 g graphite	Mortar and pestle, 30 minutes	Not attempted	Charred cake
9	10 g DC QF-6-7009 0.2 g aluminum powder, 9 g No. 1 talc	Mortar and pestle, 30 minutes	Not attempted	Powder, no char
10	10 g DC QF-6-7009 fluid 9 g No. 1 talc 0.2 g copper powder No. 132	Mortar and pestle, 30 minutes	Not attempted	Charred, slightly caked
11	6 g DC QF-6-7009 fluid 4 g No. 1 talc	Mortar and pestle, 30 minutes	Not attempted	Greasy, slightly charred appearance

TABLE III

OPEN CRUCIBLE SCREENING OF FILLERS AND FLUIDS

Formulation No.	Composition	Compounding Method	Open Crucible 600° F, 24 Hours	Open Crucible 500° F, 24 Hours	Nonspecification Brass Fittings 600° F, 24 Hours
12	30 g DC 710 fluid 10 g No. 1 talc 1 g Cab-O-Sil M-6	Mortar and pestle, 30 minutes	Not attempted	No visible change	Not attempted
13	30 g DC 710 fluid 10 g No. 1 talc 1 g Alon C	Mortar and pestle, 30 minutes	Slight drying on surface	No visible change	Assembly torque 50 ft lb, release torque 30 ft lb, no discoloration or thread distortion, sealant dried slightly
14	30 g DC 710 fluid 10 g No. 1 talc 1 g carbon black Vulcan XC-72-12	Mortar and pestle, 30 minutes	Not attempted	No visible change	Not attempted
15	30 g DC 710 fluid 2.5 g Cab-O-Sil	Mortar and pestle, 30 minutes	Slight drying on surface	Slight drying on surface	Not attempted
16	30 g DC 710 fluid 5 g Alon C	Mortar and pestle, 30 minutes	Not attempted	Slight drying on surface	Not attempted
17	30 g DC 710 fluid 3.5 g carbon black Vulcan XC-72-R	Mortar and pestle, 30 minutes	Slight drying on surface	No visible change	Assembly torque 50 ft lb, release torque 30 ft lb, no discoloration or thread distortion, sealant dried slightly
18	30 g DC 710 fluid 2 g Cab-O-Sil 5 g No. 1 talc	Mortar and pestle, 30 minutes	Slight drying on surface	No visible change	Assembly torque 50 ft lb, release torque 30 ft lb, no discoloration or thread distortion, sealant dried slightly
19	30 g DC QF-258 fluid 2.5 g Cab-O-Sil 1 g No. 1 talc	Mortar and pestle, 30 minutes	Not attempted	Soft, but some drying throughout	Not attempted
20	30 g DC QF-258 fluid 2.5 g Alon C 5 g No. 1 talc	Mortar and pestle, 30 minutes	Not attempted	Soft, but some drying throughout	Not attempted
21	35 g GE fluid 81705 3 g Cab-O-Sil 2 g No. 1 talc	Mortar and pestle, 30 minutes	Thickened, some fuming after 24 hours	Not attempted	Not attempted
22	30 g GE fluid 81705 4 g carbon black Vulcan XC-72-R	Mortar and pestle, 30 minutes	Thickened, some fuming after 24 hours	Not attempted	Not attempted

TABLE IV

SOLID LUBRICANTS AS THICKENERS AND FILLERS

Formulation No.	Composition	Compounding Method	Open Crucible 600° F, 24 Hours
23	30 g DC 710 fluid 30 g moly sulfide	Mortar and pestle, 30 minutes	Smoked in 16 minutes hardened in 3 hours
24	30 g DC 710 fluid 30 g moly sulfide technical fine	Mortar and pestle, 30 minutes	Smoked in 6 minutes, hardened in 3 hours
25	30 g GE fluid 81705 38 g moly sulfide pure	Mortar and pestle, 30 minutes	Smoked in 6 minutes, hardened in 3 hours
26	30 g GE fluid 81705 35 g moly sulfide technical fine	Mortar and pestle, 30 minutes	Smoked in 6 minutes, hardened in 3 hours
27	30 g DC QF-258 fluid 40 g moly sulfide pure	Mortar and pestle, 30 minutes	Smoked in 6 minutes, hardened in 3 hours
28	30 g DC QF-258 fluid 40 g moly sulfide technical fine	Mortar and pestle, 30 minutes	Smoked in 6 minutes, hardened in 3 hours
29	30 g DC 710 fluid 2.5 g Cab-O-Sil 1 g moly sulfide technical fine	Mortar and pestle, 30 minutes	Slight smoking after 15 minutes, some increase in viscosity after 24 hours
30	30 g GE fluid 81705 2.5 g Cab-O-Sil 1 g moly sulfide technical fine	Mortar and pestle 30 minutes	Smoked after 15 minutes, hardened in 24 hours

that at least two (and quite possibly all) of these routes were involved in base fluid decomposition. Screening of several nontoxic oxidation and polymerization inhibitors appeared to offer a means of extending the high temperature life of the fluid. Open-crucible tests were made using a commercially available inhibited methyl phenyl silicone (Dow Corning 710R) and with normal fluids to which an inhibitor was added. These experiments are recorded in Table V.

4. Indanthrene Dyestuff

A successful extreme temperature grease consisting of methyl phenyl silicone thickened with an indanthrene dye suggested an additional means to extend the extreme temperature life of the sealant.[10,11] The dyestuff, Helio Fast Blue RSV presscake, was supplied as a cake containing 22 per cent by weight dye and 78 per cent water. Efforts to compound a sealant directly from this material were unsuccessful.

TABLE V

FORMULATIONS INCLUDING OXIDATION AND POLYMERIZATION INHIBITORS

Formulation No.	Composition	Compounding Method	Open Crucible 600° F, 24 Hours
31	15 g DC 710R fluid 1.5 g Cab-O-Sil 1 g No. 1 talc	Mortar and pestle, 30 minutes	Acrid smoke in 10 minutes viscosity increase in 24 hours
32	15 g DC 710R fluid 1.5 g Cab-O-Sil 1 g No. 1 talc 0.01 g hydroquinone	Mortar and pestle, 30 minutes	Acrid smoke in 10 minutes viscosity increase in 24 hours
33	15 g DC 710 fluid 1.5 g Cab-O-Sil 1 g No. 1 talc	Mortar and pestle, 30 minutes	Fuming, no odor in 10 minutes; no fuming, viscosity increase in 24 hours
34	15 g DC 710 fluid 1.5 g Cab-O-Sil 1 g plumbous oxide 0.01 g hydroquinone	Mortar and pestle, 30 minutes	Fuming, no odor in 10 minutes; no fuming, viscosity increase in 24 hours
35	15 g DC 710 fluid 1.5 g Cab-O-Sil 1 g plumbous oxide	Mortar and pestle, 30 minutes	Fuming, no odor in 10 minutes; no fuming, viscosity increase in 24 hours
36	30 g DC 710 fluid 2.5 g Cab-O-Sil 2 g No. 1 talc 1 g aluminum powder washed†	Mortar and pestle, 30 minutes	Slight fuming, some drying in 24 hours
37	30 g DC 710 fluid 2.5 g Cab-O-Sil 2 g No. 1 talc 1 g copper powder	Mortar and pestle, 30 minutes	Slight fuming, some drying in 24 hours

†Aluminum powder washed with acetone to remove stearic acid coating.

A stock solution of the dyestuff was prepared by ball milling 50 grams of the presscake with 200 grams of Dow Corning 710 fluid for 72 hours. The water was removed by application of partial vacuum followed by increasing infrared heating in vacuo until no further moisture was evolved. The solution thus contained approximately 5 per cent (by weight) dyestuff. This solution will be entered in the remainder of this report as "5 per cent dye solution." Preliminary evaluation of formulations containing this inhibitor indicated more satisfactory fluid life at high temperature than had been obtained in earlier trials. These data are presented in Table VI.

5. Levels of Included Solids

The tendency of these formulations to thin on long standing and to separate on heating under pressure suggested that larger quantities of thickener and solid lubricant would produce a significant improvement in both sealing and antiseize properties. A series of more viscous formulations was prepared to explore the maximum permissible level of each of several solid components.

TABLE VI

FORMULATIONS INCLUDING 5 PER CENT DYE SOLUTION
(Formulations 40-47 Differ only in Amount of 5 Per cent Dye Solution and DC 710 Fluid. Each Contains 0.5 g Cab-O-Sil and 1.6 g No. 1 talc.)

Formulation No.	Composition	Compounding Method	Open Crucible 600° F, 24 Hours	Bomb Test 2000 PSIG Oxygen, 600° F	Release Test
40	1 g 5% dye solution 9 g DC 710 fluid	Mortar and pestle, 30 minutes	No visible thickening or drying, no fuming after 6 hours	Not attempted	Not attempted
41	0.5 g 5% dye solution 9.5 g DC 710 fluid	Mortar and pestle, 30 minutes	No visible thickening or drying, no fuming after 12 hours	Not attempted	Not attempted
42	0.25 g 5% dye solution 9.75 g DC fluid	Mortar and pestle, 30 minutes	No visible thickening after 12 hours	Not attempted	Not attempted
43	2 g 5% dye solution 8 g DC 710 fluid	Mortar and pestle, 30 minutes	No visible thickening or drying no fuming after 3-1/2 hours	Not attempted	Not attempted
44	3 g 5% dye solution 7 g DC 710 fluid	Mortar and pestle, 30 minutes	No visible thickening or drying, no fuming after 3-1/2 hours	Not attempted	Not attempted
45	4 g 5% dye solution 6 g DC 710 fluid	Mortar and pestle, 30 minutes	No visible thickening or drying, no fuming after 8 hours	Not attempted	Not attempted
46	5 g 5% dye solution 5 g DC 710 fluid	Mortar and pestle, 30 minutes	No visible thickening or drying, no fuming after 8 hours	Not attempted	Not attempted
47	2.5 g 5% dye solution 7.5 g DC 710 fluid	Mortar and pestle, 30 minutes	No visible thickening or drying, no fuming after 5 hours	Not attempted	Not attempted
48†	32.5 g 5% dye solution 97.5 g DC 710 fluid†† 9 g Cab-O-Sil†† 33 g No. 1 talc††	Ball mill, 24 hours	No visible thickening or drying, no fuming after 3 hours	Leakage after 30 minutes, brass and steel threads distorted	Unsatisfactory

†A portion of this preparation was forwarded to Wright Air Development Center as an initial preliminary sample.

††The final one-gram quantities of both Cab-O-Sil and No. 1 talc were added with an additional 24 hours ball milling when the sealant was observed to thin on standing.

Several filler materials not previously screened were employed to reduce separation of the liquid and solid phases in the sealant. These trials are reported in Table VII.

None of the solid materials incorporated as thickeners, fillers, lubricants, antioxidants, or polymerization inhibitors exhibited any useful property not predictable from literature describing the product.[12]

As a thickener, pyrogenic silica (Cab-O-Sil) produced a compound less susceptible to drying and caking than did finely divided alumina (Alon C) or a heat-resistant furnace black (Vulcan XC-72-R). Shear strength and lubricity of the base fluid remained unchanged.

Finely divided talc (No. 1 Lo Micron) proved to be the most satisfactory of the filler materials screened, although it did not completely overcome the separation tendency of the liquid and solid phases. Fuller's earth (Florigel SPL, DICALITE) showed reduced tendency to separate but lacked the solid lubricant properties necessary to prevent thread seizing, as did metal powders and indanthrene dyestuff. Solid lubricants (molybdenum disulfide and graphite) failed to prevent separation and decomposition of the liquid phase.

As an oxidation inhibitor, an indanthrene dye (Helio Fast Blue RSV press-cake) proved more satisfactory than did aluminum powder. It was not satisfactory in preventing phase separation or as a solid lubricant when used as the sole thickener and filler under 2000 psig oxygen at 600° F. Aluminum powder became an abrasive under these conditions.

Thickening of some of the liquids screened indicated polymerization in the base fluid. In an attempt to overcome this effect, hydroquinone and copper powder, both well known in the literature of organic chemistry as free radical inhibitors, were included in several formulations. No beneficial effects were observed with hydroquinone. Samples containing copper, which is also widely used as a catalyst for cleavage of chemical bonds, showed complete decomposition.

Of the two materials screened as solid lubricants, molybdenum disulfide proved more satisfactory. Samples containing graphite showed lesser antiseize properties than did those formulated with molybdenum disulfide. This behavior agrees with the known absorption of oxygen between the layers of graphite and formation of "graphitic acids" under extreme conditions. The level of solid lubricant was restricted by the inability of these materials to prevent separation and decomposition of the liquid phase.

C. Test Assemblies

Testing under 2000 psig oxygen at 600° F was carried out in the bomb assembly shown in Figure 2 of MIL-T-5542B(ASG), Military Specification, Thread Compound, Antiseize and Sealing, Oxygen Systems. This assembly consists of mated steel, brass, and aluminum aircraft fittings in a variety of sizes. A muffle furnace was used for heating. Incorporation of a needle valve between the gauge and the oxygen tank and incorporation of a small surge chamber adjacent to the valve formed a closed system.

TABLE VII

SCREENING OF MORE VISCOUS FORMULATIONS

Formulation No.	Composition	Compounding Method	Open Crucible 600° F, 24 Hours	Bomb Testing 2000 PSIG Oxygen 600° F, 24 Hours
49	20 g DC 710 fluid 1.8 g Cab-O-Sil 0.01 g moly sulfide†	Mortar and pestle, 30 minutes	Noticeable drying	Not attempted
50	20 g DC 710 fluid 1.8 g Cab-O-Sil 2.5 g No. 1 talc 0.01 g moly sulfide	Mortar and pestle, 30 minutes	Noticeable drying	Not attempted
51	20 g DC 710 fluid 7 g Cab-O-Sil 5 g No. 1 talc	Mortar and pestle, 30 minutes	Noticeable drying	Not attempted
52	20 g DC 710 fluid 2 g Cab-O-Sil 1 g No. 1 talc	Mortar and pestle, 30 minutes	Noticeable drying	Not attempted
53	20 g DC 710 fluid 2 g carbon black Vulcan XC-72-T 1.2 g moly sulfide	Mortar and pestle, 30 minutes	Charred, hard cake	Not attempted
54	30 g DC 710 fluid 29 g Cab-O-Sil 15 g No. 1 talc	Ball mill, 72 hours	Dry, crumbled	Failed after 5 minutes
55	30 g DC 710 fluid 29 g Cab-O-Sil 20 g No. 1 talc	Ball mill, 72 hours	Partial separation of liquid followed by excessive drying	Not attempted
56	30 g DC 710 fluid 2 g Cab-O-Sil 20 g No. 1 talc 2.7 g moly sulfide	Ball mill, 72 hours	Partial separation of liquid followed by excessive drying	Failed in 10 minutes, little, if any, thread distortion, satisfactory release
57	20 g DC 710 fluid 1 g Cab-O-Sil 3 g Dicalite White filler 0.02 g moly sulfide	Mortar and pestle, 30 minutes	Slight drying	Not attempted
58	20 g DC 710 fluid 1 g Cab-O-Sil 49 g Florigel SPL 0.029 g moly sulfide	Mortar and pestle, 30 minutes	Slight drying	Failed in 7 minutes, thread distorted, unsatisfactory release
59	20 g DC 710 fluid 1 g Cab-O-Sil 2 g No. 1 talc 3 g Florigel SPL 0.029 g moly sulfide	Ball mill, 24 hours, 30 minutes	Surface drying only	Not attempted
60	40 g DC 710 fluid 30 g Florigel SPL	Ball mill, 48 hours	Surface drying only	Held 2000 psig overnight at 75° F, standard bomb failed in 5 minutes at 600° F, all-aluminum bomb lost 500 psig oxygen pressure in 18 hours at 600° F. Aluminum fittings in both assemblies broke when bombs were dismantled. Sample recovered as white, dry powder after 24 hours at 600° F under oxygen. Unsatisfactory release.

†All of the molybdenum disulfide used in sample No. 49 and succeeding samples is "technical fine" grade.

TABLE VII (Continued)

SCREENING OF MORE VISCOUS FORMULATIONS

Formulation No.	Composition	Compounding Method	Open Crucible 600° F, 24 Hours	Bomb Testing 2000 PSIG Oxygen 600° F, 24 Hours
61	10 g 5% dye solution 30 g DC 710 fluid 2 g Cab-O-Sil 30 g Florigel SPL .04 g moly sulfide	Ball mill, steel bar, 24 hours	Dry, crumbled	Pressure dropped in 10 minutes at 600° F, fittings released easily, no thread distortion, sample partly discomposed, satisfactory release
62	40 g DC 710 fluid 3 g Cab-O-Sil 20 g Florigel SPL 0.04 g moly sulfide	Ball mill, 24 hours, add final 1.0 Cab-O-Sil, and ball mill additional 24 hours	Very slight drying	Not attempted
63	40 g DC 710 fluid 4 g Cab-O-Sil 25 g Florigel SPL 5.04 g moly sulfide	Ball mill, 72 hours, adding Cab-O-Sil gradually	Caked residue	Not attempted
64	40 g DC 710 fluid 4 g Cab-O-Sil 30 g Florigel SPL 0.04 g graphite	Ball mill, 72 hours, adding Cab-O-Sil gradually	Slight drying	Not attempted
65	40 g DC 710 fluid 4 g Cab-O-Sil 30 g Florigel SPL 5.04 g graphite	Ball mill, 72 hours, adding Cab-O-Sil gradually	Caked residue	Extensive pressure drop at room temperature. Not heated
66	40 g DC 710 fluid 4 g Cab-O-Sil 30 g Florigel SPL 0.02 g graphite	Ball mill, 49 hours, adding Cab-O-Sil gradually	Slight drying	Pressure dropped after 5 minutes, unsatisfactory release
67	40 g DC 710 fluid 2 g Cab-O-Sil 30 g Diluex 0.02 g graphite 0.02 g moly sulfide	Ball mill, 48 hours	Slight drying	Not attempted
68	40 g DC 710 fluid 2 g Cab-O-Sil 0.02 g graphite 0.02 g moly sulfide	Ball mill, 24 hours	Slight drying	Pressure dropped after 2 hours, residue powdery, fittings distorted. Unsatisfactory release
69	40 g DC 710 fluid 2 g Cab-O-Sil 20 g Diluex 10 g Aluminum (washed) MD3100 0.02 g graphite 0.02 g moly sulfide	Ball mill, 24 hours	Not attempted	Pressure dropped after 6 hours. Caked residue, fittings seized and fractured on release. Unsatisfactory release
70	40 g DC 710 fluid 1.5 Cab-O-Sil 37.5 g No. 1 talc 0.25 g moly sulfide	Ball mill, 5 days, gradually adding talc as absorbed	Very slight drying	Pressure dropped after 6 hours, white caked residue. Satisfactory release
71†	2.0 g 5% dye solution 38 g DC 710 fluid 37.5 g No. 1 talc 0.25 g moly sulfide	Ball mill, 8 days, adding talc as needed to stiffen sealant	No visible change	Pressure dropped after 1-1/2 hours. Caked residue, satisfactory

†Sample 71 was exposed to 2000 psig oxygen at 500° F. No pressure drop was observed for 12 hours. Bomb assembly was dismantled after 24 hours at 550° F. Fittings released readily and showed no thread distortion. Sample was recovered as a tan, caked material. A larger lot of this composition was submitted to WADC as a second preliminary sample.

To facilitate rapid screening of sealants, the thermocouple connection was omitted. This approximation technique appeared to be justifiable, as any vigorous combustion would be indicated even more readily by a sharp rise in the pressure of the small, closed system.

The unlike coefficients of thermal expansion of the aluminum, brass, and steel materials in the bomb assembly restrict its usefulness at 600° F. This difficulty was indicated by gross leakage from the assembly at elevated temperatures and disappearance of this leakage when the apparatus was again cooled to room temperature. This leakage was not encountered in a bomb assembly made entirely of aluminum, although the sealant used in the trial failed, and the fittings fractured on release. The aluminum fittings in all cases lost their anodized coatings on heating and tended to fracture under the specified assembly torques. Most of the high temperature leakages observed occurred about the sleeve AN819-2D, the nut AN818-2D, and the nipple AN816-2D. In view of the inability of any of the formulations tested to overcome this difficulty in the small bomb assembly, no pressure tests at 600° F were made in the larger release assembly.

As there was a delay in obtaining delivery of specification parts, early release testing was accomplished with commercial high pressure tube fittings. Neither brass nor aluminum fittings withstood the specified assembly torques without fracture. Release torques after heating in air for 24 hours at 600° F were generally about 60 per cent of the torque applied for assembly of the threaded fittings. Section 4.2.21.4 of the specification MIL-T-554B(ASB) permits release torques 25 per cent in excess of those required for assembly. A formulation was deemed to have exhibited satisfactory antiseize properties on meeting two qualifications:

1. Release torque after heating less than that applied in assembling the fittings.

2. No visible galling or thread distortion on the disassembled fittings as examined under a 40-power binocular microscope. These trials are summarized in Table VIII.

D. Formulation of Thread Compound

A stock suspension of indanthrene dye (Helio Fast Blue RSV presscake, 22 per cent) was prepared by placing 30 grams of the presscake (22 per cent dyestuff and water) in a jar mill with 120 grams Dow Corning 710 fluid and 1/2-inch metal balls. The mixture was milled continuously for 72 hours. The water was removed from this mixture by evaporation under increasing vacuum followed by gradually increased infrared heating in vacuo until no further moisture was evolved. A 120-gram portion of this suspension was transferred to the jar mill in which the final formulation was to be prepared.

To the dye suspension were added 2280 grams Dow Corning 710 silicone fluid and 90 grams Cab-O-Sil M-6. This mixture was milled for 24 hours with 2 pounds of 3/4-inch steel balls. Fifteen grams of molybdenum disulfide and 300 grams of No. 1 Lo Micron talc were added to the mixture, and milling was continued. Additional No. 1 Lo Micron talc was added in portions of not more than 500 grams at intervals of not less than 24 hours until a total of 2280 grams of talc was reached. Ball milling was continued for 72 hours after the final addition of talc.

TABLE VIII

RELEASE TESTING WITH NONSPECIFICATION, HIGH PRESSURE,
THREADED FITTINGS AFTER EXPOSURE TO AIR AT 600° F FOR 24 HOURS

Formulation No.	Thread DIA (Inch)	Material M	Material F	Applied Torque (Ft Lb)	Release Torque (Ft Lb)	Remarks
17	1/2	Brass	Brass	50	---	Fitting broke during assembly
17	5/8	Brass	Brass	65	---	Fitting broke during assembly
17	3/4	Brass	Steel	83	19	Oily film on threads after heating, no discoloration or marring of threads
	7/8	Steel	Steel	103	70	Oily film on threads after heating, no discoloration or marring of threads
17a†	5/8	Brass	Brass	65	20	Oily film on thread after heating, no discoloration or marring of threads
17a†	3/4	Brass	Brass	83	40	Oily film on threads after heating, no discoloration or marring of threads
18††	1/2	Brass	Brass	50	---	Fitting broke during assembly
	5/8	Brass	Brass	65	20	Oily film on threads after heating, no discoloration or marring of threads
22	3/4	Brass	Brass	83	10	Oily film on threads after heating, no discoloration or marring of threads
22	7/8	Steel	Steel	103	50	Oily film on threads after heating, no discoloration or marring of threads

Note: As release trials were without exception characterized by complete seizure and extensive thread damage or by a torque noticeably less than that required for assembly and no thread damage, antiseize properties of the remainder of the formulations were scored simply as satisfactory or unsatisfactory.

† A second portion of sample 17 was exposed in an open crucible at 600° F for 24 hours before application to the fittings. (Sample 17a.)

†† Sample No. 18 was exposed to air at 600° F for 24 nours before application to threads.

The resulting compound was homogeneous in appearance and had the consistency of a heavy grease.

III. DISCUSSION OF EXPERIMENTAL RESULTS

The antiseize and sealing compounds formulated in this study were designed to meet as nearly as possible the specifications of MIL-T-4452B(ASG) as amended to require prolonged exposure to 2000 psig oxygen at 600° F rather than at 160° F and under surge conditions at 744° F rather than at 302° F. Development of a material capable of complying fully with these specifications proved impossible due to the shortcomings of base materials now available on the chemical market. Experimental effort was therefore directed toward exploration of a larger number of formulations, utilizing the best materials available rather than exhaustive testing of compounds known to be inadequate. It is believed that the compound ultimately submitted for approval represents the nearest approach to complete compliance possible with available materials, and that development of a completely satisfactory compound must await further advances in the chemistry of base fluids.

Several of the sealants showed satisfactory release characteristics after exposure to 2000 psig oxygen at 600° F for 24 hours, but in no case was the compound recovered from the bomb in an unchanged condition. Easy release of the fittings in the bomb assembly could be attributed to the solid lubricant remaining on the thread after decomposition of the base fluid. The sample inside the bomb was recovered as a readily crumbled caked material at the end of 24 hours. Bomb samples held under 2000 psig oxygen at 600° F for shorter periods showed deterioration of the liquid phase increasing with time of exposure. There is no reason to suspect rapid combustion of the compound during any interval of the heating period. Inability of the sealant to prevent pressure drops at 600° F may be attributed to the varying thermal coefficients of expansion of the several metals used in the bomb fittings.

Formulation No. 71 described in Table VI of this report represents the most nearly suitable compound developed in this study. The levels of thickener, filler, oxidation inhibitor, and solid lubricant dispersed in the base fluid are those experimentally determined to be the maximum permissible without sacrifice of fluid life or of the properties contributed by one of the other components. This compound, though not serviceable at 600° F, has shown promising antiseize and sealing properties at 550° and should be useful at temperatures significantly in excess of the 302° F maximum temperature cited in the unamended Specification MIL-T-5542B(ASG).

IV. CONCLUSIONS

This study was initiated to develop a thermally stable antiseize and thread-sealing compound. Substantial progress was made in this direction, although a compound meeting all requirements was not prepared.

The following components appear to offer the greatest resistance to oxidative, thermal, and hydrolytic decomposition among the commercially available materials in their respective classes:

1. Base fluids. Dow Corning 710 fluid, a methyl phenyl silicone, showed greater resistance to decomposition than any other fluid tested.

2. Solid lubricants. Molybdenum disulfide appears to be a stable lubricant under the environment specified in this contract.

3. Thickeners. Cab-O-Sil M-6 is a satisfactory thickener.

4. Fillers. Finely divided talc imparts resistance to hardening as well as some lubricity.

5. Oxidation inhibitors. An indanthrene dyestuff retards decomposition of the base fluid and serves as part of the thickener necessary in the compound.

Decomposition of the base fluid is the principal obstacle to the preparation of a suitable compound.

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VI. APPENDIX

Commercial Products Used in the Study

DC QF-6-7009	Silicone fluid Dow Corning Corporation
GE 81644	Silicone fluid General Electric Company
Versilube, F-50	Silicone fluid General Electric Company
GE 81705	Silicone fluid General Electric Company
DC QF-258	Silicone fluid Dow Corning Corporation
DC 200	Silicone fluid Dow Corning Corporation
DC 710	Silicone fluid Dow Corning Corporation
DC 710R	Silicone fluid Dow Corning Corporation
Microfyne Graphite	Graphite John H. Dixon Crucible Co.
No. 1 Lo Micron	Talc Whittaker, Clark, and Daniels, Inc.
Micro-Cel E	Synthetic calcium silicate Johns-Manville Corporation
MD 3100	Aluminum powder Metals Disintegrating Co.
Copper lining 132	Copper powder Cresent Bronze Company
Cab-O-Sil M-6	Silicon dioxide Godfrey L. Cabot, Inc.
Vulcan XC-72-R	Furnace black Godfrey L. Cabot, Inc.
Alon C	Alumina Godfrey L. Cabot, Inc.

Helio Fast Blue RSV Presscake	Indanthrene dyestuff
Moly Sulfide (Pure)	Molybdenum disulfide Climax Molybdenum Company
Moly Sulfide (Technical fine)	Molybdenum disulfide Climax Molybdenum Company
Dicalite White Filler	Diatomaceous silica Great Lakes Carbon Company
Florigel SPL	Fuller's earth The Floridin Company
Diluex	Fuller's earth and alumina The Floridin Company