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# Welding of Ceramics

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13. ABSTRACT

All known work on the welding of refractory ceramics is reviewed and summarized. Oxides, carbides, and borides, as well as graphite, have been welded to themselves or to some metals (usually refractory metals) by either solid-state or fusion welding, or both. Of the two techniques employed, solid-state, or diffusion, welding is conducted below the melting point where sintering, diffusion, and/or reaction phenomena lead to bonding. This is generally done by means of pressure applied across the joint, and often with a fine powder in the seam. This technique will generally give the finest weld grain size, and hence higher strengths (e.g., 50,000 psi or more in $Al_2O_3$). But the total elimination of porosity can be very difficult and thus limit the use of this technique for applications in which a fraction to a few percent porosity is detrimental (e.g., to optical transmission properties). On the other hand, fusion welding is accomplished by the solidification of molten edges of the parent material or of a filler using electron-beam, optical (laser or arc-image furnaces), arc (for conductors like most carbides and borides), and flame torches, as with metals. This gives moderate (20,000-30,000 psi in $Al_2O_3$) to good (40,000-60,000 psi in some carbides and borides) weld strengths and is potentially the most economical welding method for many purposes. Other relative limitations and merits of diffusion and fusion welding are discussed along with some of the work needed to apply this technology on a broader scale.

| 14. KEY WORDS | LINK A | | LINK B | | LINK C | |
|---|---|---|---|---|---|---|
| | ROLE | WT | ROLE | WT | ROLE | WT |
| Ceramic bonding | | | | | | |
| Ceramics | | | | | | |
| Ceramic composites | | | | | | |
| Pressure welding | | | | | | |
| Arc welding | | | | | | |
| Electron beam welding | | | | | | |
| Gas tungsten arc welding | | | | | | |
| Oxyacetylene welding | | | | | | |
| Laser welding | | | | | | |
| Inert gas welding | | | | | | |
| Weldability | | | | | | |
| Weld defects | | | | | | |
| Butt welds | | | | | | |
| Solidification | | | | | | |
| Surveys | | | | | | |

# NRL-MB-20B: AN IMPROVED INSTRUMENT BALL BEARING LUBRICANT

## I. INTRODUCTION

In the latter days of World War II, this Laboratory initiated a research program that resulted in the introduction of the first ester-based turbine engine and instrument lubricants. This program has continued, directed towards the solution of broad range lubricant problems relating to Navy applications. One significant problem has been the need to improve the useful life and reliability of prelubricated instrument ball bearings used in electro-mechanical components of Navy weapons systems. Short bearing life results both from migration of the lubricant from the bearings, and from the use of instrument oils of variable or inadequate quality. Problems arising from migratory loss of lubricant are being satisfactorily solved through use of low surface energy fluoropolymer barrier coatings on outer bearing surfaces to prevent spreading of the oils away from the lubricated surfaces (1). Poor quality lubricants which shorten bearing life by excessive volatilization, and development of gummy or other unacceptable degradation products in storage or use, have not yet been eliminated. This report summarizes progress to date on the research relating to a new improved instrument ball bearing lubricant; it provides information both on the design of the oil, and on the back-up laboratory data required to formulate a comprehensive military specification which will assure the quality and performance required of the lubricant.

## II. BACKGROUND

The specification that defines the requirements for currently-used instrument oils, MIL-L-6085A (2), was originally intended for a general purpose low-temperature lubricant, one not envisioned to operate under particularly stringent conditions. Resistance to corrosion and oxidation, for example, is measured at only 121.1°C (250°F), and ASTM evaporation loss at 98.9°C (210°F). Such relatively mild conditions do not realistically examine the lubricant under the high stress conditions of thin films exposed to the heat and active metal surfaces frequently encountered in gyro, and synchro/servo bearings operating in the temperature range -55° to 150°C (-67° to 302°F) in aircraft, and 20° to 175°C (68° to 347°F) in submarines. MIL-L-6085A-qualified formulations using lighter base stocks to meet low-temperature requirements (many low-torque devices will not start up satisfactorily if the lubricant viscosity exceeds 14000 cS) may perform unacceptably at the high temperatures encountered in service.

Specification MIL-L-6085A does not take into account the ultra-cleanliness requirements of miniature ball bearings, some of which have clearances of the order of only 10 to 20 x $10^{-6}$ inches - in gyro spin motors for example. The workmanship requirement of the specification is described in general terms only, and it does not adequately rule out particulate matter of dimensions which could adversely affect operation of the bearings. The low temperature stability requirement of the specification is also seriously deficient in that generation of a "dense cloud" is specifically permitted when the oil is held for 72 hours at -53.9°C (-65°F).

Lubricants qualifying under Specification MIL-L-6085A can thus include a rather broad spectrum of quality; marginal oils may lack the stability, low volatility or cleanliness, essential to their use in miniature bearings. The problem of the variable quality of these materials is further complicated by the fact that manufacturers of rotating mechanisms are permitted to select proprietary lubricants empirically. In the case of a non-specification lubricant, neither the component manufacturer nor the Navy is protected against an unpublicized change in the lubricant formulation. On the other hand, designation by the component manufacturer of a specific proprietary lubricant from among the supposedly equivalent qualified products on the MIL-L-6085A QPL is often impractical because the designated oil may not be available at all times. The widespread use of proprietary lubricants (QPL or otherwise) has created serious logistic supply problems at distribution and repair depots; it has been tolerated by the services, however, because of the diversity of end uses of the components involved, and the absence of any satisfactory all-purpose instrument lubricant.

As part of this Laboratory's long-range effort to improve ball bearing reliability, the lubricant designated NRL MB-20B and described in the following sections was developed for the Special Projects Office for use in fluoropolymer barrier-coated ball bearings in servo/synchro applications; the subject lubricant has undergone extensive evaluation in submarine applications, and is now a required component of such systems. The Naval Ammunition Depot, Crane, Indiana, has performed successful rigorous testing of the lubricant-barrier system in the All Attitude Reference Gyro component of the Lear-Siegler AJB-3 system, and in other critical instrument component manufactured by the Bendix Company (Montrose Division).

It appears from the field evaluation data obtained to date that the subject lubricant incorporates the levels of high-temperature stability, cleanliness, corrosion protection and torque characteristics required for instrument miniature ball bearings. Specific details of these properties are given in the following sections.

## III. LUBRICANT PROPERTIES

### A. Composition and Preparation of NRL MB-20B

Military lubricant specifications have historically been based on performance rather than composition; in general this procurement philosophy has been successful. The NRL formulation MB-20B, based on a neopentylpolyol ester and a dibasic acid ester, was arrived at from considerations of both the physical and chemical properties required of the lubricant, and the availability of the constituent components. The composition of the lubricant (Batch 6A, from which many of the data reported here were obtained) is as follows:

| | Weight % |
|---|---|
| Bis(2-Ethylhexyl) Azelate[a] | 63.3 % |
| Pentaerythritol Tetracaproate[b] | 34.5 |
| Barium Dinonylnaphthylene Sulfonate[c] | 1.0 |
| Alkylated Phenylalphanaphthyl Amine[d] | 1.0 |
| Benzotriazole | 0.2 |

[a] Emery Industries
[b] Average Analysis. Hercules Powder Co. "Hercolube A"
[c] R. T. Vanderbilt Co. "NA-SUL-BSN"
[d] Geigy Chemical Company, Antioxidant "LO-6"

Slightly modified proportions (ca ± 1 or 2 percent) of the two major ester components were required (principally to meet low temperature viscosity limits) when different procurement batches of the esters were employed in the formulation. To reduce the concentrations of contaminant acids and other polar impurities which might be present, the esters were percolated through activated alumina and Florisil before incorporation into the formulation. The sulfonate rust inhibitor, supplied as a 50 percent concentrate in a light petroleum solvent, was added to sufficient percolated azelate to make about a 50 percent concentrate after the solvent was removed by stripping; an analysis for barium gave the precise final concentration of the salt. Benzotriazole was included in the formulation as a metal deactivator, used in conjunction with the amine antioxidant. The finished lubricant was passed through a 0.45 micron filter to reduce solid contaminants to an acceptable level. It should be pointed out that the most common type of filter material for removal of small particles in the micron range, cellulose esters, are chemically incompatible with synthetic ester-base lubricants. Glass fiber filters are compatible, but small fibers tend to break off and pass into the filtrate. Filters of nylon or regenerated cellulose are unaffected by esters and are considered acceptable.

Table 1 lists the important properties of the subject formulation and compares them with those of MIL-L-6085A, or other specifications where appropriate. The properties are discussed briefly.

B. Color, Cleanliness and Acidity

ASTM Union color, appearance and precipitation number of the formulation were all within MIL-L-6085A limits. As indicated earlier, the specification has no specific particle size limitation. Despite the acid number of 0.96 in NRL MB-20B, the formulation was not corrosive.

C. Viscometric and Low Temperature Properties

Over the temperature range -53.9°C to 98.9°C (-65°F to 210°F), NRL MB-20B, though slightly more viscous than a MIL-L-6085A lubricant, was nevertheless well within the requirements for low torque instrument startups (14,000 cS max). Pour point of the formulation was below -57°C (-70°F) and no turbidity or gelation developed after storage for 72 hours at this temperature.

D. Evaporation Loss and Flash Point

As was indicated in a previous section, the ASTM evaporation loss of MIL-L-6085A is specified for 98.9°C (210°F) only - an unrealistically low temperature for synchro/servo ball bearing use. Evaporation losses for the NRL lubricant were determined, therefore, at 176.7°C (350°F) for 6-1/2 hours and 22 hours. The weight losses, 6.4 and 18 percents respectively, were relatively small and were proportional to residence times in the evaporation cells. The high flash point of the NRL formulation, 221°C (430°F), relative to that of the MIL-L-6085A requirement, 185°C (365°F)(min), is yet another significant indication that the current specification tolerates use of excessively volatile base stocks.

E. Protection and Corrosivity

Instrument lubricants are required to perform satisfactorily under highly humid conditions. Rust and other corrosion products in miniature ball bearings with very close tolerances cannot be tolerated since they are destructive to both the bearing metal and the lubricant; barium dinonylnaphthalene sulfonate was therefore included in the formulation as a rust preventive. The resistance of bearing steels to rusting or corrosion when covered with a film of oil is measured in MIL-L-6085A by their performance in "protection" and "corrosivity" tests. In the former, five steel panels, pre-dipped in the oil, are exposed to 100 percent relative humidity at 48.9°C (120°F) for 100 hours; the appearance of pitting or corrosion in more than one specimen is considered unsatisfactory. In the corrosivity test, three steel discs in contact with brass clips are coated with oil and then exposed to

26.7°C (80°F) and 50 percent relative humidity for 10 days; pitting or corrosion of more than one disc is unsatisfactory. As shown in Table 1, the NRL formulation provided rust-resistant films under both conditions for double the test periods required in MIL-L-6085A. The degree of corrosion inhibition afforded by one percent of the barium salt thus appears to be acceptable.

F. Corrosion-Oxidation

MIL-L-6085A oils are required to be oxidatively stable at 121°C (250°F) for 168 hours in the presence of cadmium-plated steel, copper, steel, aluminum and magnesium. The oxidation stability of the NRL formulation was evaluated under more stringent conditions, 176.7°C (350°F). The coupons employed - copper; silver; and mild, 410 and 52100 steels - were more representative of the metals with which the oil would be in contact during service than are those in MIL-L-6085A. Changes in the oil and metals were noted after 72 hours of exposure to the test temperature. The results, listed in Table 2, indicate that the corrosion-oxidation stability of the formulation at the higher temperature was completely satisfactory with respect to viscosity and acidity changes, effect on the metals, and absence of deposits or turbidity.

## IV. THIN FILM EVAPORATION

Studies now in progress at NRL strongly suggest that thin lubricant films such as are present in miniature ball bearings, when exposed to oxygen, heat and large areas of active metal surfaces, deteriorate at faster rates and probably by different mechanisms than do bulk oils. Degeneration of thin films is evidenced by the formation of resinous products and/or granular residues, and is generally (but not invariably) accompanied by relatively large oil weight losses. No one of the properties of the NRL formulation already discussed, nor perhaps even a combination of these properties, can usefully predict the life expectancy of such films, although all make some contribution.

A simple experiment was devised which provided a reasonable measure of the stability of lubricants under thin film conditions. Between 0.3 and 0.4 gm of the liquid is contained in stainless steel planchets (5 cm diameter and one cm deep - obtainable as a commercial stock item). The samples are held for 6-1/2 hours in a thermostated, gravity convection oven at 190.4°C (375°F); weight losses and final appearance of the oils are noted. The results for NRL MB-20B are shown in Table 2 where they are compared with a phenothiazine-stabilized dioctyl adipate, a common lower molecular weight ester component of commercial MIL-L-6085A formulations. The relative weight losses were 22 and 90 percent respectively; no extraneous material was generated in the NRL-MB-20B sample, but some lacquer was formed in the adipate residue. These results are in accord with successful field experience with the NRL formulation in miniature bearings, and explain, at least in part, the poor results frequently obtained in these same bearings with marginal quality MIL-L-6085A oils.

## V. RECOMMENDATIONS

From the discussion of the foregoing data, it would appear that the NRL MB-20B prototype formulation is an acceptable basis for an improved instrument lubricant. Alternate formulations, based either on a single ester or a different combination of esters having the high performance capability of NRL MB-20B, are undoubtedly possible. There may, however, be some advantage in a formulation of the NRL type, i.e., a mixture of a higher-boiling polyol ester and a lighter diester, in that although the diester component could eventually volatilize under high-temperature operating conditions, the polyol ester component will remain in sufficient quantity to provide a liquid lubricant film. In contrast, a single-component equivalent lubricant would be more likely to evaporate completely under the same conditions.

There appears to be no need for a nonfoaming requirement for the lubricant. Under no circumstances, however, should silicone defoaming additives be permitted, because of their deleterious effect on the lubricant-bearing system.

While no attempt has been made in this NRL study to elucidate all of the technical requirements for the specification (whether this would be a revision of MIL-L-6085A or a new specification designation is an administrative matter under the purview of NAVAIRSYSCOM), the more important physical and chemical properties desired of the lubricant have been delineated. Based on the results of this study, it is recommended that the parameters listed in Table 3 be considered as target criteria for the specification. As additional laboratory and field experience is gained with the lubricant, these parameters can be modified as required.

Insufficient performance data are available to assure that, by themselves, the physical and chemical requirements proposed in Table 3 will screen out unacceptable lubricants, since only one or two formulations of the NRL MB-20B type have undergone extensive and acceptable field evaluation. If candidate oils for qualification differ in composition from this formulation, there may be some justification for including a bearing performance requirement in the specification.

ACKNOWLEDGMENTS

The authors acknowledge gratefully the major contribution to this work of their late colleague, Charles M. Murphy, who was responsible for much of the research on the physical-chemical requirements of the NRL MB-20B lubricant. The corrosion tests on NRL MB-20B were performed at the Aero Materials Department, NADC, Johnsville, Warminster, Pennsylvania, with the helpful cooperation of Mr. M. Devine and his colleagues.

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2. Military Specification MIL-L-6085A, "Lubricating Oil, Instrument, Aircraft, Low Volatility"

Table 1

Properties of NRL MB-20B

| Property | NRL MB-20B | MIL-L-6085A Requirements |
|---|---|---|
| ASTM Union Color | 4.5 | 5.0 (max) |
| Appearance | Pass | Clear, bright, transparent |
| Acid No. | 0.96 | No requirement |
| Particulate matter | <0.45μ | No specific requirement |
| Viscosity, cS | | |
| 98.9°C (210°F) | 3.54 | 3.2[a] |
| 54.4°C (130°F) | 8.75[a] | 8.0 (minimum) |
| 37.8°C (100°F) | 14.4 | 12.9[a] |
| -53.9°C (-65°F) | 12800 | 12000 (maximum) |
| Pour Point | <-57°C (-70°F) | -57°C (maximum) |
| Low Temperature Stability | -57°C (for 72 hours) | -53.9°C (for 72 hours)[b] |
| ASTM Evaporation Loss, % | | |
| 98.9°C for 22 hours | - | 1.0 (maximum) |
| 176.7°C (350°F) for 6-1/2 hours | 6.4 | - |
| 176.7°C for 22 hours | 18. | - |
| Flash Point | 221°C (430°F) | 185°C (365°F) (minimum) |

[a] Interpolated or extrapolated value
[b] Dense cloud permitted, but not gelation.

Table 2

Rust Resistance and Oxidation Stability of NRL MB-20B

| | NRL MB-20B | MIL-L-6085A Requirement |
|---|---|---|
| Protection | No failures in 100 hours. One failure in 200 hours. | Not more than 1 panel of 5 shall fail after 100 hours at 48.9°C (120°F), 100 % RH |
| Corrosivity | All 3 discs satisfactory after 20 days. | Two of 3 discs shall be free of corrosion after 10 days at 26.7°C (80°F), 50% RH |
| Corrosion-Oxidation Resistance at 176.7°C for 72 hours | | [a] |
| Viscosity change, % at 37.8°C | +5.0 | ±5% at 54.5°C (maximum) |
| Neut No change | +0.76 | ±0.5 (maximum) |
| Metal Wt. Change, mg/cm² | | |
| Copper | <0.1 | Copper ±0.2 (maximum) |
| Silver | <0.1 | Aluminum alloy ±0.2 (maximum) |
| Mild Steel | <0.1 | Mild Steel ±0.2 (maximum) |
| 410 Steel | <0.1 | Magnesium alloy ±0.2 (maximum) |
| 52100 Steel | <0.1 | Cadmium-plated steel ±0.2 (maximum) |
| Final Sample Appearance | No insolubles or gum; clear | No insolubles or gum; clear. |
| Thin Film Stability (0.3-0.4 gm sample) 6-1/2 hrs. at 190.4°C (375°F) | | |
| Weight Loss, % | 22 | 90[b] |
| Appearance | No lacquer, sludge or particulate matter. Liquid residue. | Lacquer, some liquid |

[a] Corrosion-oxidation test conditions - 121°C (250°F) for 168 hours.
[b] No requirement in MIL-L-6085A. Dioctyl adipate sample containing 1% phenothiazine.

Table 3

Target Limits for Instrument Oil Specification
Based on Experience with NRL MB-20B[a]

| Property | Proposed Requirement |
| --- | --- |
| Appearance | Clear, bright |
| Color, ASTM, Union | 5.0 (maximum) |
| Particulate Matter | 0.5 microns (maximum) |
| Acid No. | Report |
| Viscosity, cS | |
| 98.9°C (210°F) | 3.45 (minimum) |
| 37.8°C (100°F) | 14.0 (minimum) |
| -53.9°C (-65°F) | 13000 (maximum) |
| Pour Point | -57°C (-70°F) (or lower) |
| Low Temperature Stability | -57°C (72 hours) |
| ASTM Evaporation Loss, Percent 6-1/2 hours at 176.7°C (350°F) | 10. (maximum) |
| Flash Point | 210°C (410°F) (minimum) |
| Protection | Not more than 1 panel of 5 shall fail after 200 hours. |
| Corrosivity | Not more than 1 disc of 3 shall show evidence of corrosion in 20 days. |
| Corrosion-Oxidation 176.7°C (350°F) for 72 hours | |
| Viscosity Change, % at 37.8°C (100°F) | -5 to +15 |
| Neut No Change | +1.5 (maximum) |
| Metal Wt. Change, mg/cm$^2$ | |
| Copper | ±0.4 |
| Silver | ±0.2 |
| Mild Steel | ±0.2 |
| 410 Steel | ±0.2 |
| 52100 Steel | ±0.2 |
| Final Metal Appearance | No pitting or corrosion. No soft sludge. |
| Final Oil Appearance | No sludge or other insolubles. Some lacquer on cell walls permitted. |
| Thin Film Stability (sample weight 0.35 ± 0.05 gm) 6-1/2 hours at 190.5°C (375°F) (gravity convection oven) | |
| Weight loss, % | 30. (maximum) |
| Appearance | No lacquer, sludge or particulate matter. Residue is nontacky liquid. |

[a] Unless otherwise indicated, the test methods described in Specification MIL-L-6085A are applicable.