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AD

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II EFFECTS IN MULTI-PURPOSE SOAP THICKENED GREASES

TECHNICAL REPORT

By

Max T. Fisher

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U. S. ARMY WEAPONS COMMAND

ROCK ISLAND ARSENAL
RESEARCH & ENGINEERING DIVISION

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Max. T. Fisher
Research Laboratories

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ABSTRACT

To determine if additives in greases have effects upon bearing fatigue life overriding thickener and oil effects, greases of the Military Specification MIL-G-10924B type were made, with and without additives, using low viscosity naphthenic or paraffinic mineral oils thickened with calcium or lithium 12-hydroxystearate. They were tested in the rolling contact adaptation of the Four Ball Extreme Pressure Tester with 52100 steel balls at 1,200,000 psi. Hertz load and ambient temperatures.

Fatigue life improvement over the oils was greater using calcium soap instead of lithium soap as the grease thickener. Nonylphenoxyacetic acid was relatively ineffective for improving fatigue life in all greases except the lithium base paraffinic oil grease.

The insoluble crystalline additives with good lubricating properties, molybdenum disulfide and molybdenum dibutyldithiocarbamate, were very effective for improving fatigue life in greases of both thickener types. The oil miscible molybdenum dibutyldithiophosphate in the calcium base paraffinic oil grease was nearly as effective as the latter, which was the best fatigue inhibiting additive tested, but was less effective than either of the above additives in the lithium soap paraffinic oil grease. With the less effective tricresyl phosphate the relative improvement of fatigue life in these two greases was reversed.

FOREWORD

This work was performed under a project assigned to this Research Laboratory under the title, "Lubricants, Friction and Wear," Project Order 1C024401A107 AMCMS Code 5025.11.801, Sub-Task "Effects of General Types of Lubricant Additives Upon Bearing Metal Fatigue."

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PROBLEM

The purpose of this work is to determine the effects of additives in Military Specification MIL-G-10924B type greases on the fatigue life of 52100 steel ball bearings relative to the effects of the base oils and thickeners.

BACKGROUND

One of the missions of this Research Laboratory is the maintenance of Military Specification MIL-G-10924B and the qualification and surveillance of greases under its requirements. This is the most widely used grease purchased by the Government most of which goes for automotive vehicle lubrication. The extended relubrication intervals being recommended by automotive manufacturers means that lubricated parts are inspected much less often and metal fatigue becomes a more important consideration in the performance life of bearings.

Considerable work has been done on factors affecting bearing fatigue life. These factors include load, temperature, speed, surface finish, lubricant base stock and viscosity as well as metallic grain size, fiber orientation and hardness obtained by various melting and quenching techniques on many bearing compositions.(1) When this project was initiated the literature contained few references concerning the effect of lubricant additives.

A previous report on work done at Rock Island Arsenal indicated that additives in oils of the types used in Military Specification MIL-G-10924B greases have varying effects upon the fatigue life of 52100 steel balls in a rolling contact adaptation of the Four Ball EP Tester.(2)

(1) Bisson, E. E. and Anderson, W. J., "Advanced Bearing Technology," National Aeronautics and Space Administration SP-38, pp. 383-448, U. S Government Printing Office, 1964.

(2) Fisher, Max T., "Lubricant Additive Effects Upon Bearing Metal Fatigue: I.Rolling Contact Adaptation for Four Ball EP Tester," Rock Island Arsenal Research & Engineering Division Technical Report 66-1293 (AD No. 634112), April 1966.

A more recent paper by Rounds[3] states that some long chain polar additives and sulfur containing additives gave maximum beneficial life in the one to two percent region in oil whereas some chlorine and phosphorus additives were detrimental at nearly all concentrations. He believes these effects to be chemical rather than physical in nature and his data indicates that the beneficial effects may be due to the formation of surface coatings which have the proper characteristics.

It would be expected that grease thickeners would have effects upon the fatigue life of bearings. It is not known if additives in grease would have effects overriding those of these thickeners.

APPROACH

Greases of the Military Specification MIL-G-10924B type were made by the in situ reaction of either calcium oxide or lithium hydroxide with a commercial fatty acid consisting of 85% 12-hydroxystearic acid. Both the naphthenic and paraffinic oils, used in the previous investigation of bearing metal fatigue effects of oil-additive mixtures, were thickened with either the lithium or calcium soap. These were made in such a manner as to obtain approximately the same consistency at a ten percent thickener concentration. The naphthenic and paraffinic oils had viscosities of 80 SUS at 100°F. and viscosity indexes of 30 and 95, respectively.

Two additives which, in the oils, were most influential in their effects upon fatigue life in the previous work, were incorporated in five percent concentrations into one-third portions of each of the four greases. These were nonylphenoxyacetic acid, a rust preventative additive which decreased the fatigue life of 52100 steel balls, and molybdenum disulfide with which good improvement of fatigue life was obtained. No additive was included in the other one-third portion of each grease.

These greases and the base oils were evaluated in the rolling contact adaptation of the Four Ball EP Tester under the same conditions (600 kg. load at 1800 r.p.m. and ambient temperatures) as were the oil-additive combinations tested previously. This load is equivalent to approximately 1,200,000 psi. between the top and bottom balls. Thus, the fatigue effects of the oil, thickener and additive components of the grease could be compared.

(3) Rounds, Fred G., "Some Effects of Additives on Rolling Contact Fatigue," ASLE Preprint No. 66LC-27, Oct. 1966.

The test assembly was thoroughly cleaned in equal parts of chloroform, benzene and acetone before each test. Twelve tests to failure were made with each lubricant. The order of testing this series of lubricants was reversed after duplicate runs on each to compensate for differences that could be attributed to wear of the race and minimize possible effects to the race surface due to the previous lubricant used. It was necessary to use a second race adapter to obtain the 12 runs with each lubricant when wear and fatigue on the first race was obviously contributing to lower fatigue lives. The 12 tests per lubricant represent six tests with each race. These races were identical, metallurgically and dimensionally, as nearly as could be determined, but previous work occasionally showed that fatigue life can be affected by the choice of race adapters. No such pattern could be detected in this case, however. A vibration monitor and control automatically shut off the machine when a fatigue failure occurred.

The fatigue test results were treated according to statistical methods described by Leonard G. Johnson(4) except that lower ball failures were so infrequent (less than two percent) that they were disregarded and the tests repeated. The stress cycles to failure were plotted on Weibull probability paper and the bearing fatigue lives compared on the basis of 50 percent probability of survival (designated "B_{50} Life" by bearing engineers). Too few tests were run on each lubricant to get a reliable estimate of the B_{10} Life (90% probability of survival).

Excellent fatigue life improvement obtained with molybdenum disulfide as the lubricant additive in grease instigated tests using another insoluble solid anti-wear additive, molybdenum dibutyldithiocarbamate, to see if perhaps this improvement can be attributed to solid lubricants, as such. Another molybdenum compound, molybdenum dibutyldithiophosphate, was included in this series. This additive is reported to have anti-wear and extreme pressure properties but is miscible with mineral oil and, except for the phosphate radical, is chemically similar to the carbamate. A third additive in this series, the oil-miscible tricresylphosphate, provided fatigue life improvement in the oils reported previously and may serve as a check on the effects of the phosphate radical in the former compound. The base greases, containing no additives, served as reference lubricants. Because of time limitations the naphthenic oil

(4) Johnson, L. G., "The Statistical Treatment of Fatigue Experiments," Elsevier Publishing Company, 1964.

was not included as a part of this series of lubricants.

Wear on the lower three balls was recorded as milligrams per minute weight loss in an attempt to associate this property with fatigue failure time or lubricant composition.

RESULTS AND DISCUSSION

The B_{50} fatigue lives of the lubricants using Race 13 were nearly equal to those using Race 15. Consequently, the B_{50} lives of the composites concurred closely with these results. Test results using a third race were appreciably higher, however, and were not incorporated into the reported data. This discrepancy between races is unexplained but not unusual although the race adapters were made as nearly identical as possible.

The non-additive paraffinic oil greases were the only bases for comparing fatigue test results using Race 10 and Races 13 and 15. Using Race 10 the B_{50} life with the calcium base grease was a little higher than when using Races 13 and 15. With the lithium base grease the magnitude of the B_{50} lives using these races was reversed. This is also difficult to explain but the difference is not great. In considering the change of fatigue life imparted by the additives, the choice of race adapters should also be considered.

The B_{50} lives (maximum stress cycles at which no more than 50% failures can be expected) of naphthenic oil greases in Figure 1 show the lithium 12-hydroxystearate having little influence on fatigue life. Thickening the oil with calcium soap almost doubled the fatigue life obtained with the naphthenic oil. Nonylphenoxyacetic acid had a more beneficial effect in lithium base grease than in the calcium base grease but this effect was small compared to the fatigue life improvement imparted to both greases by molybdenum disulfide.

Figures 2 and 3 show these thickeners and additives having about the same influence on fatigue life with parafinic oil lubricants except that the calcium soap was less influential in the paraffinic oil. However, the B_{50} fatigue life of 52100 steel balls with this oil was nearly double that with the naphthenic oil to begin with.

Referring to Figure 4, fatigue lives with lubricants containing molybdenum disulfide was independent of oil and thickener effects. The influence of the calcium soap and nonylphenoxyacetic acid in calcium soap greases was relatively

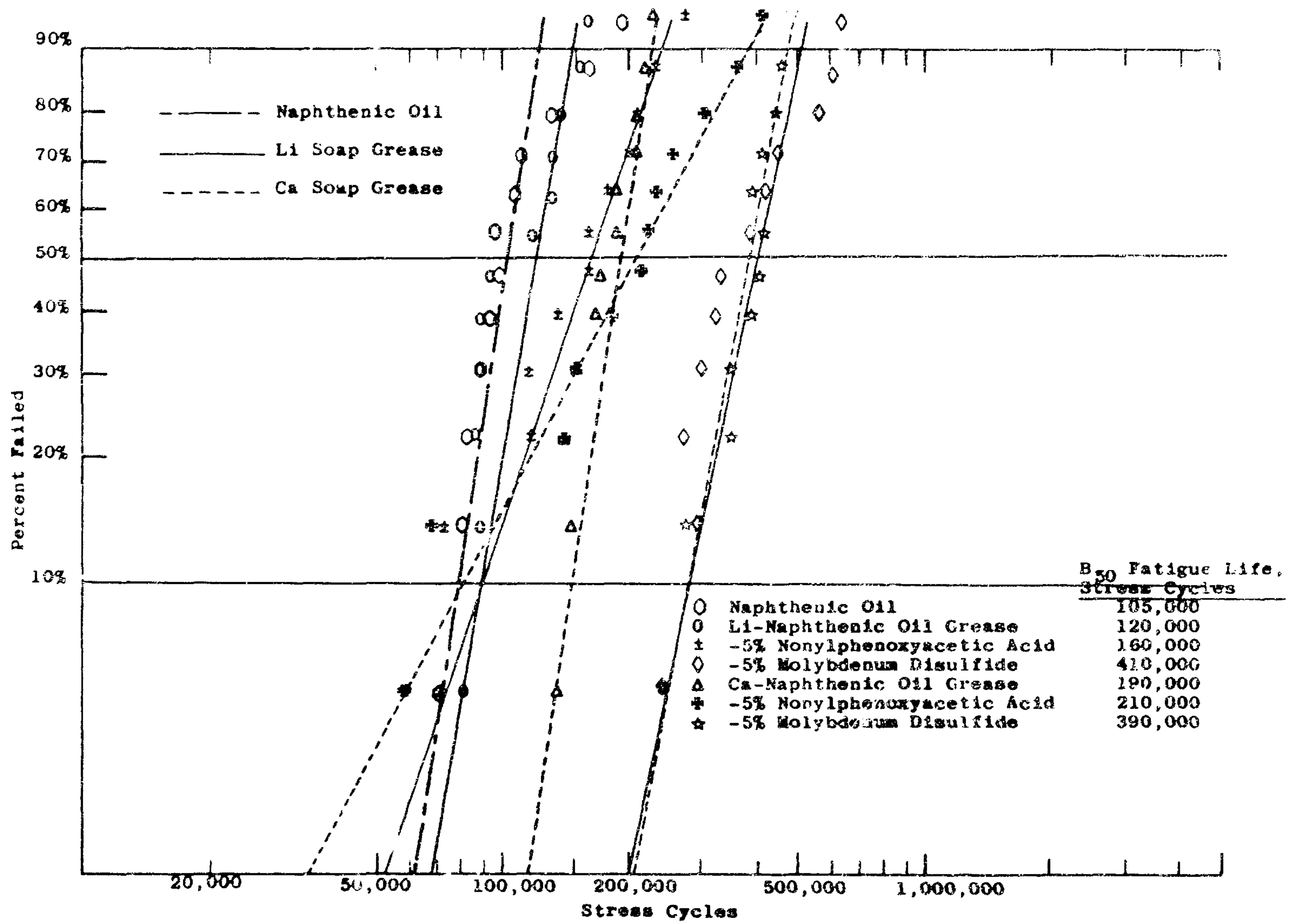

FIGURE 1 FATIGUE LIFE WITH NAPHTHENIC OIL GREASES (RACES 13 & 15)

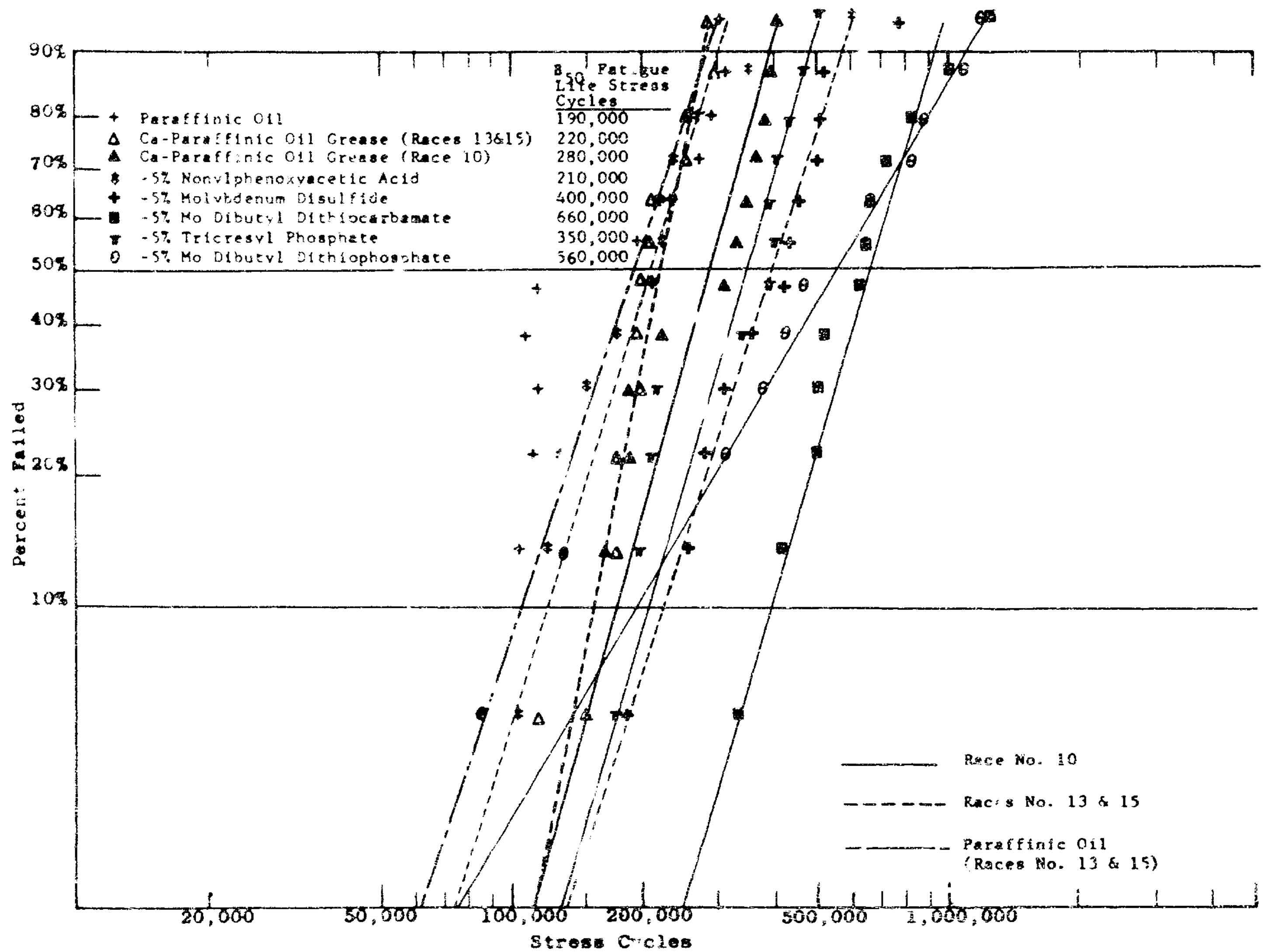

FIGURE 2 FATIGUE LIFE WITH CALCIUM SOAP-PARAFFINIC OIL GREASES

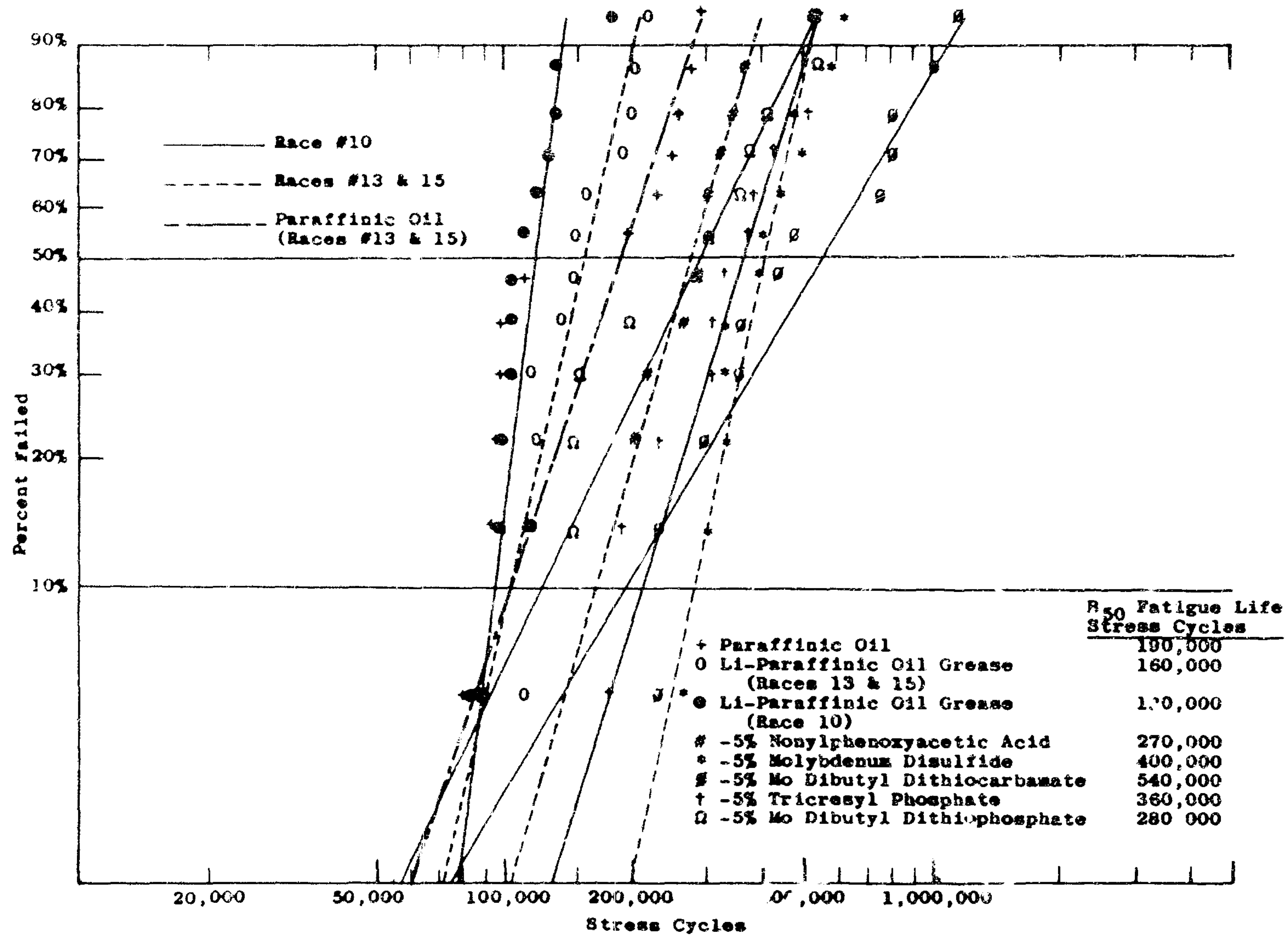

FIGURE 3 FATIGUE LIFE WITH LITHIUM SOAP PARAFFINIC OIL GREASES

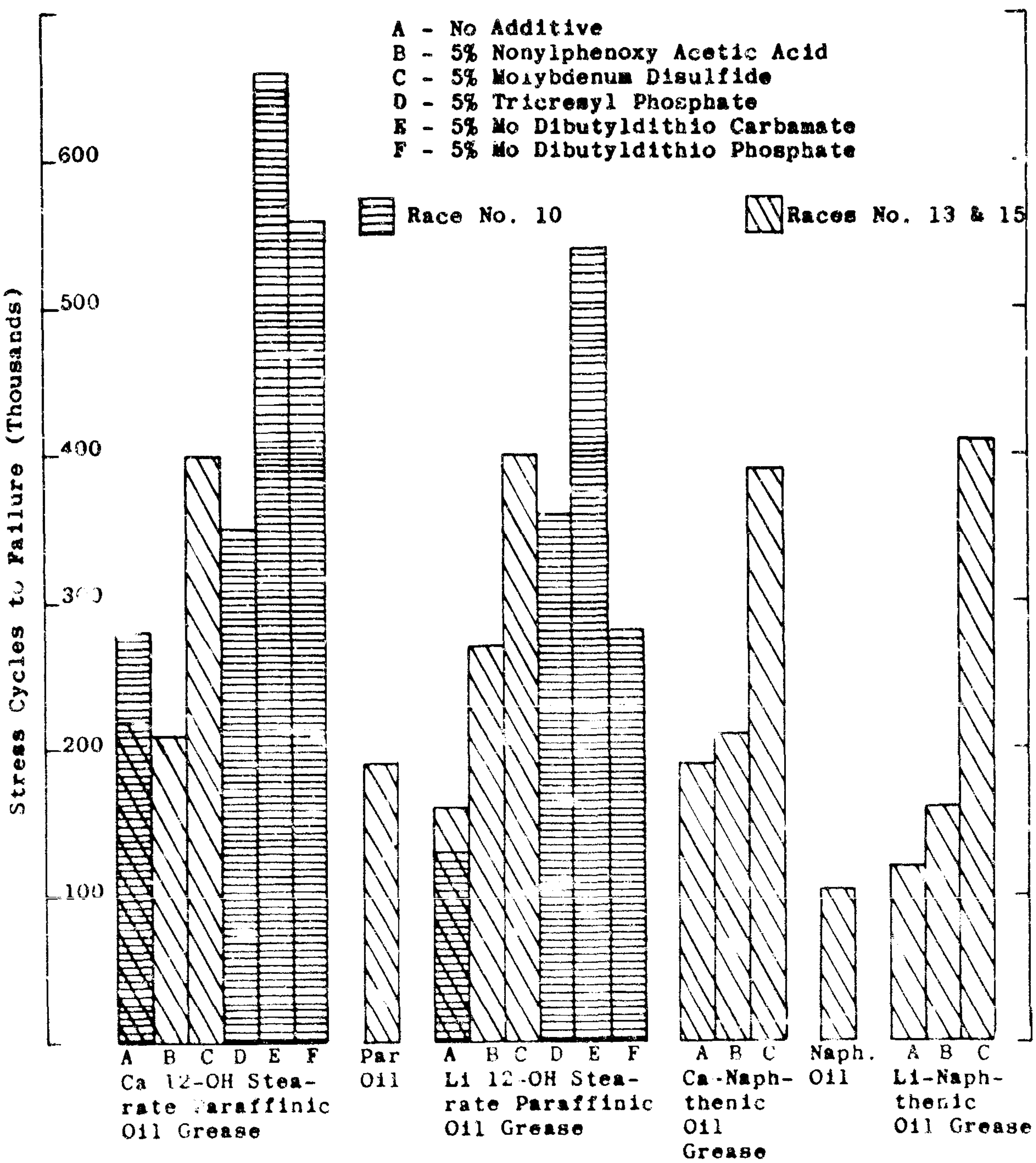

FIGURE 4 LUBRICANT ADDITIVE EFFECTS ON THE B_{50} FATIGUE LIFE OF 52100 STEEL BALLS

independent of oil influences. This was not true with lithium base greases. Tricresyl phosphate in paraffin oil greases also seemed to have an effect upon fatigue life independent of thickener type.

There appears to be an interaction between one or both soaps and the molybdenum dibutyldithiocarbamate or phosphate additives to form different surface coatings on the balls which results in varying fatigue lives. The appearance of the cleaned lower balls after test fortifies this theory. The color of these balls was quite distinctive with the latter two additives depending on whether they were incorporated into lithium or calcium base greases. No difference in ball appearance was visually detectable with the other additives.

Of primary interest is the quite pronounced improvement of 52100 steel ball bearing fatigue life with the second insoluble solid additive, molybdenum dibutyldithiocarbamate. This improvement is considerably greater than that obtained with molybdenum disulfide, which showed the most beneficial effects in the previous series of tests. Molybdenum dibutyldithiophosphate in the calcium soap-paraffinic oil grease also showed excellent fatigue life improvement but in lithium soap grease the improvement was much less.

Table I shows the wear rates on the bottom three balls of the Four Ball Fatigue Life Tests with the various lubricant compositions and their relationship to fatigue life.

Little difference in wear could be detected between lubricants containing naphthenic and paraffinic oils except possibly for the nonadditive lithium soap greases. The fatigue life, however, was generally less with lubricants containing naphthenic oil.

Calcium soap greases made with these oils reduced wear to nil and increased fatigue life. Wear reduction was less with the lithium soap thickener and the fatigue life changed little, increasing slightly in naphthenic oil and decreasing a little in paraffinic oil.

The wear and fatigue properties of three of the four greases were affected little by the addition of nonylphenoxyacetic acid. In the lithium base paraffinic oil grease, however, wear was reduced and fatigue life increased with this additive.

TABLE I

WEAR-FATIGUE LIFE RELATIONSHIP IN ROLLING CONTACT ADAPTATION OF FOUR BALL EP TESTER

	Race No.	Wear, Av. Mg/Min on Bottom 3 Balls	Fatigue Life, Minutes Average	Fatigue Life, Minutes B_{50}
Paraffinic Oil	13 & 15	0.167	50	53
-10% Calcium 12-Hydroxystearate	13 & 15	0.007	59	61
	10	0.001	79	78
-5% Nonylphenoxyacetic Acid	13 & 15	0.007	64	58
-5% Molybdenum Disulfide	13 & 15	0.005	115	111
-5% Tricresyl Phosphate	10	0.004	95	97
-5% Mo Dibutyldithiocarbamate	10	None	189	183
-5% Mo Dibutyldithiophosphate	10	None	165	155
-10% Lithium 12-Hydroxystearate	13 & 15	0.062	43	44
	10	0.156	33	36
-5% Nonylphenoxyacetic Acid	13 & 15	0.025	79	75
-5% Molybdenum Disulfide	13 & 15	0.007	117	111
-5% Tricresyl Phosphate	10	None	100	100
-5% Mo Dibutyldithiocarbamate	10	0.002	162	150
-5% Mo Dibutyldithiophosphate	10	0.017	82	78
Naphthenic Oil	13 & 15	0.172	31	29
-10% Calcium 12-Hydroxystearate	13 & 15	0.002	50	53
-5% Nonylphenoxyacetic Acid	13 & 15	0.007	60	58
-5% Molybdenum Disulfide	13 & 15	0.005	107	108
-10% Lithium 12-Hydroxystearate	13 & 15	0.036	31	33
-5% Nonylphenoxyacetic Acid	13 & 15	0.029	44	44
-5% Molybdenum Disulfide	13 & 15	0.008	11[illegible]	114

Molybdenum disulfide, tricresylphosphate and the two organic molybdenum additives had no adverse effects on wear in the calcium base greases with which wear was already very low. These additives in lithium base greases reduced wear to a minimum with one exception. This exception was that molybdenum dibutyldithiophosphate in lithium soap paraffinic oil grease reduced the wear only to a level comparable to that obtained with nonylphenoxyacetic acid in this grease. Fatigue life with the latter two greases was also nearly the same and considerably shorter than that with the other lithium soap additive combinations in paraffinic oil.

Evidently high wear rates are a factor in obtaining lower fatigue lives but not the only influence. There is quite a variation in the fatigue lives of greases showing little or no wear.

CONCLUSIONS

The rolling contact adaptation for the Four Ball Extreme Pressure Tester has proved to be a useful and relatively economical means of determining fatigue effects of lubricants on 52100 steel balls.

The low viscosity paraffinic oil was a little more beneficial to fatigue life than was the naphthenic oil of the same viscosity at 100°F. Since these tests were run at ambient temperatures, the lower viscosity of the naphthenic oil at the higher temperatures could be an influence.

Lower fatigue lives were usually accompanied by higher wear rates.

Calcium base greases were usually more beneficial to fatigue life and wear than their lithium base counterparts.

Nonylphenoxyacetic acid was relatively ineffective for improving fatigue life in all greases except lithium 12-hydrostearate-paraffinic oil grease.

The insoluble solid additives with good lubricating properties, molybdenum disulfide and molybdenum dibutyldithiocarbamate, were very effective in all greases for extending the fatigue life of 52100 steel balls.

Molybdenum dibutyldithiophosphate was very beneficial to fatigue life in the paraffinic oil thickened with calcium soap but much less effective in this oil thickened with lithium soap. For the other phosphate additive, tricresyl phosphate, the relative improvement of fatigue life in these two greases was reversed.

The color of the balls after test was distinctive with each of the organic molybdenum additives, depending on whether they were incorporated into the lithium or calcium base grease. This and varying fatigue results indicate interaction between the additives, thickeners and balls to form different surface coatings having unique properties. Fatigue results indicate this is also true with nonyl-phenoxyacetic acid but no distinctive coloration of the balls collaborates this.

The ball surface coloration and B_{50} Life was unique to the additive with molybdenum disulfide and tricresyl phosphate regardless of which grease they were incorporated into. The thickener influence was evidently nil with greases containing these additives.

RECOMMENDATIONS

It is recommended that additives normally used in Military Specification MIL-G-10924B greases be evaluated for their effects upon bearing metal fatigue singly and in combination. Also the effects on fatigue life of the insoluble solid type lubricant additives in fully formulated greases of this type should be determined. Possible adverse effects on fatigue life of otherwise excellent additives might be neutralized or obscured by the excellent fatigue extending properties of these solid lubricant additives.

The effects upon fatigue life of non-soap thickened greases should also be evaluated.

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