

UNITED STATES PATENT OFFICE

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HIGH-TEMPERATURE BALL-BEARING GREASE

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This invention relates to high temperature ball bearing greases suitable for operation at temperatures in the range 300° F. to 500° F. and upwards.

Such greases must be soft and unctuous over a wide temperature range, capable of lubricating at ordinary temperatures, and also, and of more importance, capable of maintaining a physical nature not greatly changed and capable of remaining in place to effect lubrication at high temperatures, ranging up towards 500° F.

The principal object of this invention is to provide such a grease. Other objects will be obvious in part or developed hereinafter. Among such objects may be mentioned certain improvements in nature of the grease effected by special handling methods.

This invention is based upon the discovery that such greases may be produced by compounding appropriate mineral oils with the soaps of certain fatty acid materials hereinafter more fully described, accompanied by certain compounding procedures.

The fatty acids with which this invention is particularly concerned are those fatty acids which have been separated from fish oils and have been hydrogenated under controlled conditions so as to yield a product having an iodine number between about 60 and about 170. Some particular fish oil fatty acids, or distillate cuts therefrom, are also found to have iodine numbers within this range, and the desired acid may also be gotten by making appropriate blends of any of the above noted materials. For example, certain commercial products are desirable for these purposes. A product known as "Hydrogenated fish oil, iodine No. 77," is one such product. This so-called "fish oil" (the trade name appears to be a misnomer), is a fatty acid (and not a glyceride), having an acid number of 201. It has an iodine number of 77, as indicated. Another suitable commercial product is a partially hydrogenated fish oil with a saponification number of 196, an acid number of 0.6, and an iodine number of 139.

These products, saponified to form an alkali

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metal soap and handled as hereinafter disclosed, together with a proper amount of a proper mineral lubricating oil, form the typical grease with which this invention is concerned. To this grease there may be added small amounts of other ingredients without departing from the spirit of this invention.

Small amounts of the soaps of other acids may be utilized, either as extenders or diluents of the characterizing soaps above noted, or to confer some desired or desirable change in properties of the grease. For example, a certain amount of the alkali soap of 12 hydroxystearic acid may be substituted for up to about 25% of the fish oil fatty acids, and when so used, a grease of a given consistency may be produced with a lower overall consumption of fatty material. Or, valuable changes in nature may be gotten by the addition of a small amount of the calcium soap of acetic acid or of calcium soaps of other fatty acids of up to 4 carbon atoms.

Similarly, inhibitors, such as phenyl-alpha-naphthylamine or other amines, or other known inhibitors may be added, for the usual purposes; or small amounts of water resistant soaps, such as zinc naphthenate, zinc resinate, or cadmium resinate, may be added; or extreme pressure ingredients, as certain lead soaps, may be added; all without departing from the spirit of this invention.

The mineral oil vehicle used may be any appropriate oil, paraffinic, naphthenic, or other base, ranging in viscosity from about 200 to 1500 seconds Saybolt Universal at 100° F. In the experimental data here presented, the oils used were of naphthenic type, ranging in viscosity from 500 seconds to 1500 seconds Saybolt Universal viscosity at 100° F.

These greases derive a significant characteristic both from the choice of fatty acid component and from the character of the compounding process. In the first place, choice of a fatty acid component having an iodine number of from about 60 to about 170 gives a material capable of polymerization under heating. These fatty materials, dissolved in the mineral oil compo-

ment, or in a portion of it, are saponified at temperatures of from about 180° F. to about 250° F. After saponification, the mix is raised in temperature at a controlled rate of not over about 2 degrees per minute until a temperature within the range of about 500° F. to about 540° F. is attained, and then held there until a period of from about 20 minutes to about 30 minutes has elapsed, sufficient for the unsaturated acids used to undergo polymerization.

It is necessary that sufficient time be allowed for this polymerization since undertreatment will result in a product which will be soft in penetration, of poor yield, will have a low melting point and a low operating temperature range. Also, undertreated products tend to become hard and crack during service.

After a proper polymerization, these greases

may be cooled by any of the usual methods to form greases of desirable structure, consistency, and drop point. It has been found preferable, however, to shock chill the greases, preferably by rapidly chilling in any one of several grease chilling devices which are commercially available. The desirable additional characteristics can be gotten by cooling at a rate not less than about 10 degrees per minute from the cooking temperature down to about 300° F. Below this temperature, the rate of cooling appears to be immaterial.

Exemplary of greases so made, the following tabular data is presented. In these tabulations, "Fatty Acid A" is a fish oil fatty acid having an acid number of 201 and an iodine number of 77. "Fatty Oil B" is a partially hydrogenated fish oil of saponification number 196, acid number 0.6 and iodine number 139.

TABLE I
High temperature ball bearing greases

Batch Number	1	2	3	4	5	6
Material	Percent by weight	Percent by weight	Percent by weight	Percent by weight	Percent by weight	Percent by weight
Fatty Acid A	12.0		15.0	15.0	29.0	15.0
Fatty Oil B	12.0	15.0	5.0	5.0		5.9
12-Hydroxystearic Acid		5.0	0.5	0.5	0.5	
Sebacic Acid						
Behenic Acid						
Sodium Hydroxide	5.7	4.6	5.0	5.0	6.7	4.8
Naphthenic Mineral Oil	69.2	73.9	72.5	72.5	62.6	74.2
Zinc Resinate		0.5			0.2	
Lithium Stearate						
Cadmium Resinate	0.1		1.0			
Calcium Acetate				1.0		
Barium Stearate			1.0	1.0	1.0	1.0
Oxidation Inhibitor	1.0	1.0				
Method of Cooling	Slow (pan)	Slow (pan)	Slow (pan)	Slow (pan)	Slow (pan)	Slow (pan)
Time of cooling to 300° F., Hrs.	3	3	3	3	3	3
Tests on Grease						
Penetration, Unworked	150	180	120	120	190	195
Penetration, Worked	170	200	130	130	210	215
ASTM Dropping, °F	500+	498	500+	500+	477	496
Free NaOH, Percent	0.6	0.5	0.7	1.0	0.3	0.70
Remarks	(1)	(2)	(3)	(4)	(5)	(6)

Remarks

- (1) The pan cooled product was very satisfactory—no oil leakage in the pan cooled block nor in the milled grease.
- (2) The pan cooled product was very satisfactory—no oil leakage in pan cooled block nor in the milled grease. Very good performance test.
- (3) No oil leakage in block form and none in the milled product. Very good product slowly cooled or rapidly cooled.
- (4) No oil leakage in block form nor any in milled product. Good product pan cooled or quickly cooled.
- (5) No oil leakage in block form nor any in milled product.
- (6) Pan cooled; sample seemed to bleed a lot of oil during cooling and the milled sample seemed to bleed a lot too.

TABLE II
High temperature ball bearing greases

Batch Number	7	8	9	10	11
Material	Percent by Weight	Percent by Weight	Percent by Weight	Percent by Weight	Percent by Weight
Fatty Acid A	14.5		15.0	15.0	14.4
Fatty Oil B		15.0			14.4
Stearic Acid		5.0		5.0	
12-Hydroxystearic Acid	4.9		5.0		
Behenic Acid			4.4	4.8	6.4
Sodium Hydroxide	3.1	4.6	74.6	74.2	64.8
Naphthenic Mineral Oil	77.5	74.4	1.0	1.0	
Oxidation Inhibitor		1.0			
Method of cooling	Rapid	Rapid	Rapid	Rapid	Rapid
Time of cooling to 300° F., Hrs.	0.1	0.1	0.1	0.1	0.1
Tests on Grease					
Penetration, Unworked	282	165	182	195	120
Penetration, Worked	297	175	198	215	130
ASTM Dropping, °F	432	475	474	496	509+
Free NaOH, percent	0.3	0.7	0.5	0.7	0.7
Remarks	(7)	(8)	(9)	(10)	(11)

Remarks

- (7) The product when pan cooled is very soft and bleeds a lot of oil. The product when milled from liquid state to solid results in a very nice buttery grease.
- (8) Quickly cooled and milled results in a very satisfactory grease. The pan cooled product separates into soap and oil.
- (9) Quickly cooled product is very nice upon milling and very little leakage of oil.
- (10) Very nice product when milled from liquid state to solid.
- (11) When quickly cooled nice product but not too satisfactory when pan cooled.

It will be noted that the greases shown in Table I were slowly cooled from cooking temperatures to 300° F., while the greases of Table II were shock chilled, being cooled to 300° F. in a period of 6 minutes. Greases 6 and 10, which are quite similar in proportioning, tend to point up the value of shock-chilling, since 6, when slowly cooled, tends to bleed. In general, comparison between Table II and Table I shows that with higher oil content grease, shock chilling becomes of importance. Concurrently with shock chilling, it is usual and frequently advisable to accompany the chilling with working. Since usual devices for shock chilling, such as internally scraped double pipe exchangers and the like, provide some such working, and may be changed readily to provide more, this is easy of accomplishment.

In general, in compounding such greases, the mineral oil content will vary from about 70 to about 85 percent, and the soap content derived from the indicated acids of iodine numbers from about 60 to about 170 will vary from about 30 to about 15% of the final product.

I claim:

1. An improved lubricating grease adopted for the lubrication of ball bearings, roller bearings, and the like at high temperatures comprising a major portion of a mineral lubricating oil and from about 15 to about 30 percent of an alkali metal soap of a fatty acid having an iodine number in the range of from about 60 to about 170, the grease having been compounded by incorporating the acid-containing ingredient in at least a portion of the oil, saponifying, and then raising the temperature of the mix slowly to a temperature of from about 500° F. to about 540° F. followed by holding the temperature at that level for a period of at least about 20 minutes and sufficient to bring about substantial polymerization of the fatty acid component.

2. An improved lubricating grease adopted for the lubrication of ball bearings and the like at high temperatures comprising a major portion of a mineral lubricating oil and from about 15 to about 30 percent of an alkali metal soap of a fatty acid sufficiently unsaturated to have an iodine number in the range of from about 60 to about 170, the grease having been compounded by saponification of the acid-containing ingredient in the presence of at least a portion of the oil, followed by raising the temperature of the mix slowly to a temperature of from about 500° F. to about 540° F., and holding the temperature at such level for a period of about one-half hour, followed by cooling of the resulting grease.

3. An improved lubricating grease adopted for the lubrication of ball bearings, roller bearings, and the like at high temperatures comprising a major portion of a mineral lubricating oil and from about 15 to about 30 percent of an alkali metal soap of a fatty acid having an iodine number in the range of from about 60 to about 170, the grease having been compounded by incorporating the acid-containing ingredient in at least a portion of the oil, saponifying, and then raising the temperature of the mix slowly to a temperature of from about 500° F. to about 540° F. followed by holding the temperature at that level for a period of at least about 20 minutes and sufficient to bring about substantial polymerization of the fatty acid component followed by

rapid cooling of the resulting grease to a temperature of about 300° F.

4. An improved lubricating grease adopted for the lubrication of ball bearings and the like at high temperatures comprising a major portion of a mineral lubricating oil and from about 15 to about 30 percent of an alkali metal soap of a fatty acid sufficiently unsaturated to have an iodine number in the range of from about 60 to about 170, the grease having been compounded by saponification of the acid-containing ingredient in the presence of at least a portion of the oil, at a temperature of from about 180° to about 250° F., followed by raising the temperature of the mix at a rate of about 2° F. per minute to a temperature of from about 500° F. to about 540° F., and holding the temperature at such level for a period of about one-half hour, followed by rapid cooling of the resulting grease.

5. An improved lubricating grease adopted for the lubrication of ball bearings and the like at high temperatures comprising a major portion of a mineral lubricating oil and from about 15 to about 30 percent of an alkali metal soap of a fatty acid sufficiently unsaturated to have an iodine number in the range of from about 60 to about 170, the grease having been compounded by saponification of the acid-containing ingredient in the presence of at least a portion of the oil, at a temperature of from about 180° to about 250° F., followed by raising the temperature of the mix at a rate of about 2° F. per minute to a temperature of from about 500° F. to about 540° F., and holding the temperature at such level for a period of about one-half hour, followed by rapid cooling of the resulting grease to about 300° F. at a rate not less than about 10° F. per minute.

6. An improved lubricating grease adopted for the lubrication of ball bearings and the like at high temperatures comprising a major portion of a mineral lubricating oil and from about 15 to about 30 percent of an alkali metal soap of a fatty acid sufficiently unsaturated to have an iodine number in the range of from about 60 to about 170, the grease having been compounded by saponification of the acid-containing ingredient in the presence of at least a portion of the oil, at a temperature of from about 180° to about 250° F., followed by raising the temperature of the mix at a rate of about 2° F. per minute to a temperature of from about 500° F. to about 540° F., and holding the temperature at such level for a period of about one-half hour, followed by rapid cooling of the resulting grease to about 300° F. at a rate not less than about 10° F. per minute while working said grease.

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