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LUBRICANT GREASE

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The present invention relates to novel lubricant greases and more particularly to novel greases wherein oleaginous materials are thickened to grease consistency with fluorocarbon resins.

Various types of greases have long been used in the lubrication of anti-friction bearings such as ball or roller bearings. These greases must provide a low torque and low temperature rise in the bearing and they should have long high temperature lubrication life so that frequent repacking of bearings is unnecessary.

In the past the so-called complex and soap type greases have been generally accepted as satisfactory for most applications. Soap greases are thickened with a soap-type grease thickener which contains an alkali or alkaline earth metal soap of a high molecular weight soap-forming carboxylic acid and/or petroleum sulfonic acid.

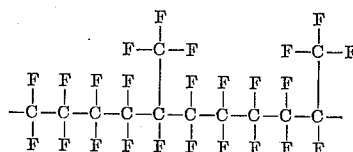
Technology has advanced rapidly in the past few years with the development of machines, engines, etc., which operate at higher speeds and higher temperatures than their forerunners. Consequently, a need has arisen for greases capable of withstanding these more severe conditions of operation. To meet this need, soap thickened greases have been improved in many respects and have been found quite useful in many high-speed, high-temperature operations.

In addition, other thickening agents have been developed in an attempt to find a grease capable of satisfactorily withstanding severe operating conditions. One of these thickening agents is a polymerized tetrafluoroethylene. Greases thickened with this polymeric material have been prepared and evaluated under various conditions of operation. In several instances such greases were found to be satisfactory and even advantageous in comparison with certain soap greases known to the prior art. However, it was observed that greases thickened with polymerized tetrafluoroethylene lost their consistency and broke down to an undesirable extent when used in applications requiring high speed and consequently high temperature machine and engine operation.

According to the present invention, it has now been unexpectedly found that a particularly advantageous grease results when an oleaginous vehicle is thickened to grease consistency with a resinous material which is the product of copolymerizing tetrafluoroethylene and hexafluoropropylene. Such a grease composition possesses unique properties making it particularly adaptable for high speed application at elevated temperatures up to 450° F. and higher. Unexpectedly greases of the present invention thickened with a fluoroethylenepropylene resin possess excellent wear, extreme pressure, and high temperature properties and retain their consistency for long periods of operation in contrast to the relatively poorer performance given by the chemically similar tetrafluoroethylene resin thickened greases. Fluoroethylenepropylene resin thickeners have been observed according to the present invention to impart superior properties to a variety of oleaginous vehicles including the hydrocarbon oils, ester oils, and silicone oils such as fluorosilicone oil.

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The fluoroethylenepropylene resin thickeners of the present invention are prepared by polymerization of tetrafluoroethylene with hexafluoropropylene to form a copolymer having the following repeating unit:



Typically, these fluoroethylenepropylene resin thickeners are in a finely divided white powder form with a particle size on the order of 10 to 15 microns. Other characteristic properties of these materials have the following values.

Table 1

Melting point	545°-565° F.
Specific heat	.28 cal./g./° C.
Refractive index:	
At 475μ	1.344±.003.
η_a^{25}	1.388.
Hardness Rockwell (ASTM 785-51T)	R25.
Water absorption (ASTM D570)	.01%.

Fluoroethylenepropylene resins such as these are available commercially and have been marketed under the trademark "Teflon 120 FEP" fluorocarbon resin. These materials are further processed for use in this invention by drying, heat treating and grinding.

The resin will be used in particular applications in an amount sufficient to thicken the oleaginous vehicle to the desired consistency and impart the desired characteristics to the resulting grease. This amount will vary with the requirements of the particular application for which a grease of the present invention is being formulated. Typically, however, a grease of the present invention will contain fluoroethylenepropylene resin thickener in an amount from about 20 to about 60 percent by weight of the total grease composition. In particular applications it may be advantageous to employ the thickener in an amount from about 25 to about 50 percent by weight, and in preferred formulations the thickener will be present in an amount from about 25 to about 45 percent by weight.

The thickeners of the present invention may be employed in conjunction with a wide variety of oleaginous lubricant vehicles. All types of mineral lubricating oils as well as synthetic oils including diesters, complex esters, hydrocarbon polymers, and silicone oils having lubricating oil characteristics, may be used. Specific oils which may advantageously be employed as the vehicle in grease formulations of the present invention include hydrocarbon base oils such as solvent extracted SAE 40 Oil and unextracted SAE 50 Oil. In addition, ester oils, such as that marketed commercially under the name "Hercolube A" and silicone polymer oils, may be used. The preferred silicone oil is a fluorosilicone oil having the following characteristics:

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Table 2

	Viscosity grade	
	300 cs.	1,000 cs.
Color.....	Water white to straw	
Viscosity, centistokes at:		
0 F.....	5,500	22,000
77 F.....	300	1,000
210 F.....	30	74
400 F.....	5.5	11.5
Specific gravity at 77°F.....	1.25	1.28
Freeze point.....	-55F	-40F
Flash point, open cup.....	500F	560F
Fire point.....	600F	620F
Volatility, weight percent:		
4 hours at 392° F.....	2.0	1.5
120 hours at 392° F.....	18.0	5.0
240 hours at 392° F.....	24.0	8.0
Gel time, hours at 392° F.....	350	350
Acid number.....	Trace	Trace
Surface tension, dynes per centimeter.....	25.7	26.1

Silicone polymer oils of this type can be obtained commercially from Dow Corning Corporation as FS 1265 fluid. Grease formulations according to the present invention may be prepared according to methods known in the art such as by colloid milling, roll milling, etc.

To emphasize the advantage to be obtained through use of greases prepared according to the present invention to contain fluoroethylenepropylene (FEP resin) resin thickeners, these materials were compared with a similar grease thickened with tetrafluoroethylene resin (TFE resin). Results of that comparison are shown in columns 1 and 2 of Table 3. It will readily be seen from comparison of results set forth therein that the grease thickened with fluoroethylenepropylene resin is equivalent to the tetrafluoroethylene resin thickened grease in all other tests and out performs the tetrafluoroethylene resin thickened grease by a factor greater than 10 in the leakage test. This difference in percent leakage is highly important because leakage is an especially significant measure of the all-important property of grease stability. The lower the leakage value, the greater the amount of grease retained within the bearing, the more uniformly the thickener is dispersed throughout the vehicle, the greater the stability of the grease and thus the higher is its efficiency.

That the fluoroethylenepropylene resin thickeners are versatile and can be adapted for use in conjunction with various oleaginous vehicles can be seen from columns 3, 4 and 5 of Table 3.

Table 3

	1	2	3	4	5	6
FEP resin, wt. percent.....	30		43	43	40	
TFE resin, wt. percent.....		30				
Lithium soap, wt. percent.....						7
Fluorosilicone oil, wt. percent.....			37			
"HERCOLUBE A" ester oil, wt. percent.....				57		
Solvent extracted SAE 40 base oil, wt. percent.....					60	93
Unextracted SAE 50 base oil, wt. percent.....	70	70				
Penetration at 77° F. ASTM D217-60T.....	333	337	207	325	288	265-295
Leakage at 210° F., percent ¹	1.2	16.5	0	2.7	1.3	4.6
4 Ball wear test scar diam., MM.....	0.67	0.71				0.97
4 Ball extreme pressure test weld pt., KGS.....	355	355				100

¹ Federal test method—standard 791 method no. 321.

When the fluorosilicone oil, the ester oil and the solvent extracted SAE 40 Oil were used in conjunction with the fluoroethylenepropylene resin thickener, the penetration remained at a sufficiently high level while leakage was kept well within the range established by the data in column 1, and in one case, that of the fluorosilicone oil, there was no leakage whatsoever. Each of these specific formulations, therefore, demonstrates a surprising and

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significant advantage over that of the tetrafluoroethylene resin thickened grease of column 2. The aforementioned grease formulations were compared with a conventional lithium soap grease having the concentrations and exhibiting the properties set forth in column 6. It will be seen that the percent of leakage is reduced in each of the fluoroethylenepropylene resin thickened greases by a factor of at least 2 in comparison with the lithium soap grease. The fluoroethylenepropylene thickened greases are clearly superior to the lithium soap greases in the other significant categories, as will be seen from a comparison of the data in column 2 with the data in column 6.

In higher temperature applications, the advantage of employing greases thickened with fluoroethylenepropylene resins in comparison with greases thickened with tetrafluoroethylene resins becomes even more apparent. Table 4 shows the results of high temperature (450° F.) operation for extended periods in the L-35 Bearing Performance Test with greases thickened with fluoroethylenepropylene resins.

Table 4

FEP resin, wt. percent.....	35	30
Silicone fluid, wt. percent ¹	65	70
L 35 test, hrs. (450° F.; 10,000 r.p.m.; 5 lbs. radial, 5 lbs. axial load).....	1,230+	1,120+

¹ Dow Corning Company DC 510 silicone fluid.

Both grease formulations were still performing satisfactorily after more than 1000 hours of operation when the test runs were voluntarily stopped. By way of contrast, satisfactory tetrafluoroethylene thickened greases for operation at these higher temperatures could not be formulated. Attempts to prepare grease formulations using the latter mentioned thickeners in conjunction with vehicles of the type reported herein failed. Formulations attempted balled-up, lost their consistency and were found to be unsatisfactory. Accordingly, no L-35 test runs were made with tetrafluoroethylene thickened greases.

The L-35 Test is Coordinating Research Council Test CRCL-35-59 developed and published by the Coordinating Research Council, 30 Rockefeller Plaza, New York 20, New York.

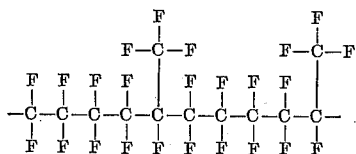
Having thus described the invention, what is claimed is:

1. A grease-type lubricant composition consisting essentially of an oleaginous lubricant vehicle selected from

the group consisting of a hydrocarbon oil, a silicone lubricating oil and a synthetic ester lubricating oil and from about 20 to about 60 percent by weight of copolymer of tetrafluoroethylene and hexafluoropropylene said copolymer having a melting point from about 545 to 565° F., a specific heat of .28 cal./g./° C., a refractive index at 475μ of about 1.344, a Rockwell hardness

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of R25 and a water absorption of .01%, and said copolymer having the following repeating unit:



2. A grease-type lubricant composition as described in claim 1 in which the lubricant vehicle is a hydrocarbon lubricating oil.

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3. A grease-type lubricant composition as described in claim 1 in which the lubricant vehicle is a silicone oil.

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