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INORGANIC GELLING AGENT THICKENED FLUOROCARBON GREASE COMPOSITIONS

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This invention relates to greases obtained from fluorocarbon oils and/or the related poly(chlorotrifluoroethylene) lubricants.

Substantially completely fluorinated hydrocarbon oils, that is hydrocarbon oils which have been fluorinated to such an extent that substantially all of the hydrogen atoms of the compounds in the oils have been replaced by fluorine atoms, are valuable as lubricants and for heat transfer media under extremely corrosive conditions, since they are stable and inert to most chemicals. Fluorinated oils and methods of preparing them are disclosed, for example, in Ind. and Eng. Chem., volume 39, No. 3, March, 1947, on pages 290–298, 319–329, 343–345 and 350–354. Such completely fluorinated hydrocarbon oils are sometimes, for convenience, referred to here as fluorocarbon oils.

A closely related class of highly inert lubricating media comprises the liquid polymers of chlorotrifluoroethylene or, as otherwise described, perfluorovinyl chloride. These lubricants also are described in the same volume of Ind. and Eng. Chem., referred to above, especially in the paper beginning on page 333. These fluid polymers may be prepared either by polymerization of the monomer to form fluid products or by cracking high molecular weight polymers to give low molecular weight materials having the characteristics of lubricating oils.

While these materials are outstanding for their high stability in the presence of corrosive agents such as concentrated nitric acid and the like, satisfactory greases made therefrom have not been prepared heretofore. Ordinary soaps are oxidized by nitric acid and are not easily compatible with these fluorinated compounds, and other known thickening agents tested hereinbefore have shortcomings giving rise to unsatisfactory products. For example, the use of gelling amounts of perfluorinated soaps results in grease compositions, but these are subject to rapid changes in consistency and viscosity with temperature. The use of polymeric thickeners, such as interpolymers of tetrafluoroethylene and chlorotrifluoroethylene of high molecular weight, likewise results in the formation of thickened oils and greases having too high a response to thermal changes to provide satisfactory greases.

Such classes of gelling agents as have been described in U. S. Patent 2,260,625 by Kistler, referred to as aerogels and similar colloidal materials are suitable for use in the preparation of greases and like materials. The utilization of amorphous inorganic gels for the preparation

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of greases entails the benefit of substantially no change in the consistency of the product with change in temperature but when placed in water the halocarbon separates free of the silica. Such materials have been improved in this respect by providing them with a hydrophobic surface, as referred to in U. S. Patent 2,584,085 to Stross, which describes the use of polymeric silanes which appear to unite chemically with the surface of the gel particles.

A further type of water-proof gelling agent exhibiting substantially little response to thermal changes comprises the onium clays described in U. S. Patent 2,531,440 to Jordan. The latter type of material, however, possesses the disadvantage of permitting the bearing to run hot and causing excessive corrosion when the bearing is water wet.

Silica may also be provided with a hydrophobic surface which is chemically bound thereto by surface esterification with alcohols having from 3 to 18 carbon atoms per molecule. This material when incorporated in a lubricating oil has the same disadvantage with respect to poor lubrication in water contaminated bearings as noted above for the onium clays.

It is an object of the present invention to provide grease compositions especially useful for under-water conditions as well as under extremely corrosive conditions. It is a second object of this invention to provide grease compositions which are resistant to the oxidizing influences of strong oxidizing agents, such as nitric acid, but which at the same time provide satisfactory lubrication for and protection of metal surfaces. It is a particular object of the invention to provide grease compositions which are resistant to the action of water and which do not rise to the surface thereof if disengaged from a metal surface while submerged in water. Other objects will become apparent during the following discussion.

Now in accordance with the present invention, it has been found that grease compositions, which accomplish the above and other objects comprise a lubricating phase which is a substantially completely halogenated hydrocarbon oil (i. e., halocarbon) wherein the halogen has a molecular weight less than 40 and wherein at least about 3 out of about every 4 halogen atoms in the molecule are fluorine atoms, said oil being gelled to a grease consistency by means of an inorganic gelling agent, the surface of which has a hydrophobic acid-resistant monomolecular surface coating preferably chemically united therewith; these greases also contain a minor amount of a

substantially completely fluorinated polar organic compound having from 3 to 18 carbon atoms per molecule.

Greases prepared which have the above-defined ingredients possess the unique set of properties of being highly resistant to attack by strong acids and oxidizing agents, such as concentrated nitric acid. Due to the preferred chemical bond between the inorganic gel surface and the monomolecular hydrophobic layer thereon, the greases are highly water resistant as well as inert toward acids and oxidizing agents. Moreover, due to the presence of the substantially completely fluorinated polar compounds (hereafter referred to as perfluoro polar compounds) the resulting greases have the surprising property of wetting metallic surfaces preferentially with respect to water, thus overcoming the previously noted disadvantages with respect to surface-esterified silica, onium clays and the other hydrophobic inorganic gelling agents more specifically defined hereinafter.

These greases are of special importance for the lubrication of metal parts of aircraft utilizing mixtures of fuels with strong oxidizing agents, such as concentrated nitric acid, as the power means for said aircraft. Lubricants known to the art heretofore have not been satisfactory for this purpose due either to their sensitivity with respect to acids or oxidizing agents, and/or lack of the proper hydrophobic properties, and shortcomings with respect to proper lubricant wetting of metallic surfaces as well as their changes in consistency upon alteration of temperature. These greases also are suitable (due to the extremely high density) for the lubrication of submarines and other submerged mechanisms, since any grease which becomes detached from the latter while it is submerged will not rise to the surface of water as do ordinary lubricants, thus disclosing to surface craft the position of the submerged vessels.

The ingredients to be used in the present compositions are all essential to the successful production of satisfactory products for the defined uses. In the absence of the perfluoro polar compounds unsatisfactory lubrication is obtained. In the case of aircraft, for example, the planes require frequent washing due to the presence of highly corrosive liquids, such as nitric acid, thus exposing the metal parts to corrosion and grease displacement. The polar perfluoro organic compounds which have been found to be especially suitable for use in these compositions comprise especially those having from 3 to 18 carbon atoms per molecule and containing polar radicals such as carboxyl, alkoxyl, aldehydo, amino, keto or ester groups.

While salts and soaps of the perfluoro acids may be utilized, it is preferred that these be employed in amounts which are small enough so that appreciable thickening of the cold compositions does not occur. That is, soaps of perfluoro fatty acids, for example, may cause the formation of grease structures similar to the action of ordinary fatty acids in mineral lubricating oil but with the inhibiting disadvantage of passing through phase transitions during temperature elevation, thus causing softening of the compositions. Consequently, while the salts and soaps of the perfluoric polar organic compounds may be used, it is preferred that they be present in amounts which do not cause any substantial thickening cold composition.

To be effective in the present compositions the perfluoro polar compounds should be utilized in amounts by about 1% and about 10% by weight based on the total composition. Preferably the aliphatic perfluoro compounds are employed and the most effective class which has been tested comprises the perfluoro fatty acids, especially those containing from 6 to 18 carbon atoms per molecule. Suitable species include the substantially completely fluorinated acids, such as butyric, valeric, caproic, caprylic, capric, lauric, meristic, palmitic and stearic acids. While the perfluorinated derivatives of the aliphatic monocarboxylic acids are preferred, other suitable classes include the perfluorinated derivatives of aliphatic dicarboxylic acids and the perfluorinated cyclic acids, including cycloaliphatic and aromatic acids. Suitable dicarboxylic acids include glutaric, adipic, pimelic, and sebacic acids.

In addition to the perfluoro fatty acids and the other organic carboxylic materials the perfluorophosphoric, phosphonic and phosphinic acids as well as their partial esters may be employed. The alcohols, particularly the fatty alcohols, which have been perfluorinated are suitable, such as perfluoro octyl alcohol, decyl alcohol, dodecyl alcohol, and the like. The corresponding ketones and aldehydes may be employed for the present purpose.

As noted hereinbefore, the lubricating oil utilized in the present compositions is a substantially completely halogenated hydrocarbon oil wherein the halogen has an atomic weight less than 40 and wherein at least about 3 out of every 4 halogen atoms in a molecule are fluorine atoms. The two principal classes of lubricating oils to which this definition applies comprise the fluorocarbons and the lower polymers of chlorotrifluoroethylene.

The substantially completely fluorinated hydrocarbon oils (fluorocarbon oils) which may be employed are those which have a distillation range at 10 mm. Hg pressure from about 50° C. to about 300° C. (about 150° C. to about 460° C. at atmospheric pressures). The term "substantially completely fluorinated" means that substantially all of the hydrogen atoms of the compounds in the hydrocarbon oils have been replaced by fluorine so that the fluorinated oil contains at least 75% chemically combined fluorine.

These fluorocarbon oils may be prepared by fluorination of hydrocarbon oils by methods known to the art and then fractionating the fluorinated oil usually at about 10 mm. mercury pressure to obtain products of desired boiling range. The hydrocarbon oils employed for preparing the fluorocarbon oils are mixtures of hydrocarbons and may be naturally occurring mineral oils, such as (paraffinic) base, napthenic base or highly aromatic petroleum oils (usually highly refined), or synthetic hydrocarbon oils of similar composition, such as the Fischer-Tropsch oils and those obtained by the platforming of paraffin waxes. Preferably, the hydrocarbon oils are composed entirely or mainly of aromatic hydrocarbons. Usually, the hydrocarbon oil is vaporized, the vapors diluted with inert gas and the mixture then passed over a fluorinating agent such as cobalt trifluoride. Representative fluorocarbon oils and methods for preparing them are disclosed in the Ind. and Eng. papers, Volume 39, referred to hereinbefore.

While fluorocarbon oil fractions distilling over the full range of from about 50° C. to about 300° C. at 10 mm. pressure may be employed, it will

generally be desirable to employ fractions distilling over narrow ranges within the broad range. It will usually be preferred to employ fluorocarbon oil fractions having distilling ranges wholly above 80° C. at 10 mm. (200° C. at atmospheric pressures) and particularly those having distilling ranges between 80° C. and about 200° C. at 10 mm. (200° C. to 400° C. at atmospheric pressure). Especially useful fractions comprise those having distilling ranges falling in the range of from about 120° C. to about 240° C. at 10 mm. pressure. The use of oils distilling below the temperature for treatment will require the use of pressure to avoid the loss of oil by vaporization during the processing or alternatively the use of excessive amounts of oil to allow for loss by vaporization. The viscosity of the fractions of fluorocarbon oil varies with their distilling range, those distilling at higher temperatures having higher viscosities.

The second principal class of suitable lubricants, comprising the polymers of chlorotrifluoroethylene, may be prepared by the polymerization of the monomer or the cracking of high molecular weight polymers to give products within the general boiling range defined hereinbefore and preferably having molecular weights between about 300 and 1500, especially between about 650 and 1000. The terminated radicals on each of the polymer chains is immaterial to the present invention but in accordance with the description of these oils given in papers appearing in volume 39, Ind. and Eng. Chem., starting at page 333, the end groups are probably $-CF_2Cl$.

The above fluorocarbon oils and the equivalent polymers of chlorotrifluoroethylene are gelled to a grease consistency by means of an inorganic gelling agent bearing a hydrophobic acid-resistant monolayer surface coating. The classes of these materials have been referred to hereinbefore. The preferred class comprises surface-esterified silica, especially where the esterifying alcohol is an aliphatic alcohol having from 3 to 18 (preferably 3–12) carbon atoms. These alcohols may be either monohydric or polyhydric, but preferably are the fatty alcohols, such as propyl, butyl, amyl, hexyl, octyl, and decyl alcohols. The proportion of alcoholic radicals chemically required to esterify the surface of the silica gel will depend to a certain extent upon the surface area of said gel. A sufficient degree of esterification should be carried out to provide a monomolecular layer of ester radicals on at least 50% of the surface of the gel when the latter has a surface area between about 100 and 1000 square meters per gram thus constituting from about 7.5% to about 15% by weight of the esterified silica. Another class of suitable gelling agents comprises the onium clays which are described in the patent to Jordan, U. S. 2,531,440. The onium clays which are suitable in the present greases comprise especially those in which the onium radical is derived from amines, although phosphonium, stiponium, silanonium compounds may be used instead. According to the Jordan disclosure the onium clays comprise onium modified clays originally exhibiting a base exchange capacity of at least 25 in which the exchangeable inorganic cation has been replaced by an onium base of the classes defined above. The classes which are especially suitable are the bentonites of the swelling type, such as Wyoming bentonite and the magnesium bentonites such as hectorite. Other possible but less suitable materials include attapulgite and Georgia-Florida

clay. Preferably, the ammonium compounds used to form onium clays are primarily or secondarily aliphatic ammonium compounds containing at least one organic radical with a linear dimension of at least 14 linear Angstrom units and preferably having at least 10 carbon atoms in the chain. The onium clays especially suitable are those having a swelling capacity in nitrobenzene of at least 8-fold. Suitable species include the ion exchange reaction product of dimethyl dicetyl ammonium bromide with Wyoming bentonite, the ion exchange reaction product of triphenyl lauryl phosphonium bromide with hectorite, and dodecyl ammonium bentonite.

In addition to the onium clay reaction products a closely similar class comprises the onium synthetic zeolites. The latter class of materials are more clearly defined as complex ammonium silicates containing exchangeable cations such as hydrogen, sodium, potassium, barium and magnesium.

Synthetic clays prepared by known methods are also suitable for the preparation of synthetic onium zeolite products including the magnesium-silica-sodium oxide type or those composed of lime, silica and potassium oxide.

Another type of colloid useful in the present compositions comprises inorganic gelling agents such as amorphous colloids wherein at least about 50% of the surface is coated with a water-proofing polymeric silicone resin. Usually, the resin is formed by polymerization of a halo-silane or an amino-silane on the surface of the colloidal gel and it is possible that the surface of the gel enters into a chemical reaction with the coating, although the exact nature of the chemical bond is at present undefined. It has been established however that products of this character are not only water-proofed but are also inert to oxidizing and acidic agents such as concentrated nitric acid. Coated gels of this character are described in Stross patent U. S. 2,584,085. The silica and other gelling agents preferably are highly porous gels such as are obtained by the preparation of a hydrogel and replacement of the water therein with the oil by solvent transfer. Suitable gelling agents other than silica which may be so coated include magnesia, alumina, and mixtures of such materials such as magnesia-silica and alumina-silica as well as the corresponding metallic oxides, hydroxides, sulfides and carbonates. The organo silicon halides are preferably those in which the organo group is a hydrocarbon radical and especially the dialkyldihalosilanes, particularly the dialkyldichlorosilanes wherein the alkyl radicals have from 1 to 6 carbon atoms each. These and other classes are exemplified by tripropylbromosilane, dimethyldichlorosilane, and diallyldichlorosilane. The corresponding amino-silanes may be employed such as ditertbutoxydiaminosilane.

The compositions containing the essential ingredients described above preferably have the following ratio of ingredients:

	Parts by weight
Perfluorocarbon oil	70–98
Hydrophobic gel	1–20
Perfluoro polar organic compound	1–10

Greases comprising the recited class of fluorocarbon oils or the analogous polymers of chlorotrifluoroethylene gelled with the hydrophobic gelling agents described above provide satisfactory lubrication in the absence of water only. They are resistant to the action of strong acids

or oxidizing agents including concentrated nitric acid, but in the presence of water which would occur frequently, the greases are displaced from the metal surfaces which they are supposed to protect and lubricate. Under these circumstances the water displacing the grease causes excessive corrosion of metallic parts and the latter, if they are moving elements such as bearings, tend to heat up during operation. The correction of these shortcomings comprises an essential feature of this invention. The addition of perfluoro polar organic compounds to the grease system not only corrects the deficient metal wetting properties of the system, but moreover, does so while still maintaining the proper inert character with respect to strong oxidizing agents and acids. The exact mechanism by which this correction occurs is not entirely clear at this time, so no theory for the action can be advanced. However, it has been found that the addition of the recited ratio of perfluoro polar organic compounds to the grease permits the bearings to run at a cooler temperature and reduce the corrosion caused by water to a negligible figure.

The use of the particular class of water-proofed gelling agents appears to be essential to the successful operation of these compositions. Other gelling agents do not appear to be satisfactory. For example, soaps are largely incompatible with the fluorocarbon oils and their equivalents and do not form proper greases therewith. Moreover, the greases which are so formed are subject to softening due to phase transitions upon heating. The use of inorganic gelling agents in their place in the absence of any hydrophobic monolayer coating is impractical, since the metal objects protected by the greases are frequently in contact with the water such as during the washing of planes or in under-water operations of submarine craft. The use of higher polymers such as high molecular weight poly(chlorotrifluoroethylene) for thickening fluorocarbons and the like has the same shortcomings as soap greases in that they show too great an effect of change in consistency with respect to temperature.

It is possible to water-proof inorganic gels such as clays and amorphous colloids including silica and the like by absorption on their surfaces of hydrophobic surface-active agents including amines, soaps, hydroxy compounds, and fatty acids. However, apparently due to the fact that these coatings are merely absorbed on the gelled surfaces and not chemically reacted therewith, they have been found to be subject to attack by strong acids such as nitric acid. As distinguished from this, the present gelling agents as described above bear hydrophobing monolayers on at least 50% of their surfaces which are chemically reacted therewith and, whether due to this or other reasons, are surprisingly inert towards strong oxidizing agents. In summary, therefore, it will be seen that the present combination of ingredients has a unique set of physical and chemical properties not heretofore attained. The examples which follow illustrate various embodiments of this invention.

Example I

A fluorocarbon lubricating oil having the trade name "Standard Fluorolube" produced by Hooker Chemical Corp. was gelled to a grease consistency with a silica aerogel. This grease performed satisfactorily in a bearing test under anhydrous conditions. However, upon addition of a small amount of water the grease structure was rapidly destroyed and bearing lubrication was immediately lost. A modification of this grease was made by replacing silica aerogel with a silica having approximately similar surface area (in the order of 600 square meters per gram) the surface being esterified with butyl alcohol so that the butylated silica contained approximately 9.5% isobutyl radical. 5% of this butylated silica was used to gel the same fluorocarbon oil. The grease which resulted was resistant to the action of nitric acid and its structure was maintained even in the presence of water. However, during a bearing operation test, when water was added the latter displaced the grease from the bearing surface and caused the bearing to run hot and also resulted in excessive bearing corrosion. This second grease was modified by the further addition thereto of 2% by weight of perfluorocaprylic acid based on the total grease composition. The modified grease was resistant to the action of concentrated nitric acid, maintained its structure in the presence of water and adhered strongly to bearing surfaces, even in the presence of water, thus protecting the bearing from hot operation and from corrosion as well.

Example II

A polymer of chlorotrifluoroethylene having a molecular weight of about 900 was gelled to a grease consistency by the presence of magnesia-alumina water-proofed by absorption of polymerized dimethyldiaminosilane. The resulting grease was resistant to the action of nitric acid and maintains its consistency in the presence of water. However, when water was added during a bearing operation test, the bearing was seriously corroded. Modification of this grease with 3% by weight of perfluorostearyl alcohol overcame the latter shortcoming and permits the bearing to operate even in the presence of water without any deleterious effect. The latter grease was likewise substantially inert with respect to nitric acid.

Example III

Similar experience was obtained with respect to greases comprising a fluorocarbon oil prepared by substantially complete fluorination of a medium viscosity lubricating oil gelled to a grease consistency with a highly expanded silica-alumina gell, the latter containing about 5% alumina. Grease structure was immediately lost upon addition of water. The gel was hydrophobed in a second experiment by absorption and polymerization of dimethyldichlorosilane on the gel surface followed by water washing to remove undesired by-products. The modified gel was coated with approximately 1 part of the polymerized organo silica halide for each part by weight of the inorganic gel to obtain a monomolecular coating on the gel surfaces. Dispersion of this product in fluorocarbon oil resulted in the formation of a grease which was not only resistant to the action of nitric acid but also maintained its structure in the presence of water. Lubrication of motor-bearings in the presence of water with this grease was not satisfactory due to displacement of the grease by the water. Addition of perfluoromethylcaproate in an amount of about 1% by weight of the grease resulted in a composition exhibiting satisfactory bearing operation even in the presence of water since the grease volume on the bearing surface was maintained.

Example IV

Suitable bearing operation can be obtained by

the use of the following composition: Poly-(chlorotrifluoroethylene), molecular weight 900, 85 parts; silica surface esterified with 10% by weight of hexyl alcohol, 10 parts; perfluorocapramide, 5 parts.

Example V

Another composition possessing the desired characteristics with respect to bearing lubrication in the presence of water is as follows: fluorocarbon lubricating oil, SAE 30 grade, 90 parts; silica surface esterified with 7.5% by weight of amyl alcohol, 8 parts; copolymer, 1 mole each of tetrafluoroethylene and chlorotrifluoroethylene, average molecular weight: 90, 1 part; heptafluorobutyronitrile, 1 part.

We claim as our invention:

1. A grease composition comprising as the lubricating oil a substantially completely halogenated hydrocarbon oil, the halogen atoms having atomic weights less than 40, the oil distilling at temperatures in the range of from about 50° C. to about 300° C. at 10 mm. Hg pressure, said oil being gelled to a grease consistency with an inorganic gelling agent bearing a hydrophobic acid-resistant monomolecular surface coating chemically united therewith, said grease containing from about 1% to about 10% by weight of a substantially completely fluorinated polar organic compound having from 3 to 18 carbon atoms per molecule.

2. A grease composition comprising as the lubricating oil a substantially completely fluorinated hydrocarbon oil distilling at temperatures in the range of from about 50° C. to about 300° C. at 10 mm. Hg pressure, said oil being gelled to a grease structure with a silica gel having its normal water content substantially entirely replaced by said oil, the surfaces of the gel being esterified with aliphatic alkoxy radicals, said radicals having from about 3 to about 12 carbon atoms each, the grease also having dispersed therein from about 1% to about 10% by weight of a substantially completely fluorinated polar organic compound having from about 3 to about 18 carbon atoms per molecule.

3. A grease composition comprising as the lubricating oil a substantially completely fluorinated hydrocarbon oil distilling at temperatures in the range of from about 130° C. to about 300° C. in 10 mm. Hg pressure, said oil being gelled to a grease structure with from about 3% to about 10% by weight of a surface-butyl esterified silica gel, the butoxy groups constituting between about 7.5 and about 15% by weight of said gel, the grease also containing from about 1% to about 5% by weight of perfluorocaprylic acid.

4. A grease composition comprising as the lubricating oil poly(chlorotrifluoroethylene) oil distilling at temperatures in the range of from about 50° C. to about 300° C. at 10 mm. Hg pressure, said oil being gelled to a grease structure with a silica gel having its normal water content substantially entirely replaced by said oil, the surfaces of the gel being esterified with aliphatic alkoxy radicals, said radicals having from about 3 to about 12 carbon atoms each, the grease also having dispersed therein from about 1% to about 10% by weight of a substantially completely fluorinated polar organic compound having from about 3 to about 18 carbon atoms per molecule.

5. A grease composition comprising as the lubricating oil a substantially completely fluorinated hydrocarbon oil distilling at temperatures in the range of from about 50° C. to about 300° C. at 10 mm. Hg pressure, said oil being gelled to a grease structure with a silica gel having its normal water content substantially entirely replaced by said oil, the surfaces of the gel being esterified with aliphatic alkoxy radicals, said radicals having from about 3 to about 12 carbon atoms each, the grease also having dispersed therein from about 1% to about 10% by weight of a substantially completely fluorinated fatty acid compound having from about 3 to about 18 carbon atoms per molecule.

6. A grease composition comprising as the lubricating oil a substantially completely fluorinated hydrocarbon oil distilling at temperatures in the range of from about 50° C. to about 300° C. at 10 mm. Hg pressure, said oil being gelled to a grease structure with a silica gel having its normal water content substantially entirely replaced by said oil, the surfaces of the gel being esterified with aliphatic alkoxy radicals, said radicals having from about 3 to about 12 carbon atoms each, the grease also having dispersed therein from about 1% to about 10% by weight of a substantially completely fluorinated fatty alcohol having from about 3 to about 18 carbon atoms per molecule.

7. A grease composition comprising as the lubricating oil a substantially completely fluorinated hydrocarbon oil distilling at temperatures in the range of from about 50° C. to about 300° C. at 100 mm. Hg pressure, said oil being gelled to a grease structure with a silica gel having its normal water content substantially entirely replaced by said oil, the surfaces of the gel being esterified with aliphatic alkoxy radicals, said radicals having from about 3 to about 12 carbon atoms each, the grease also having dispersed therein from about 1% to about 10% by weight of a substantially completely fluorinated aliphatic polar organic compound having from about 3 to about 18 carbon atoms per molecule.

8. A grease composition comprising as the lubricating oil a substantially completely fluorinated hydrocarbon oil distilling at temperatures in the range of from about 50° C. to about 300° C. at 10 mm. Hg pressure, said oil being gelled to a grease consistency by a highly expanded inorganic gel bearing a monomolecular layer on the surface thereof of an organo silicone polymer water-proofing agent, said grease also having dispersed therein from about 1% to about 10% by weight of a substantially completely fluorinated polar organic compound having from about 3 to about 18 carbon atoms per molecule.

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