Oct. 29, 1963 **R. A. BUTCOSK** 3,108,965

GREASE MANUFACTURE

Filed Aug. 22, 1960 2 Sheets-Sheet 1

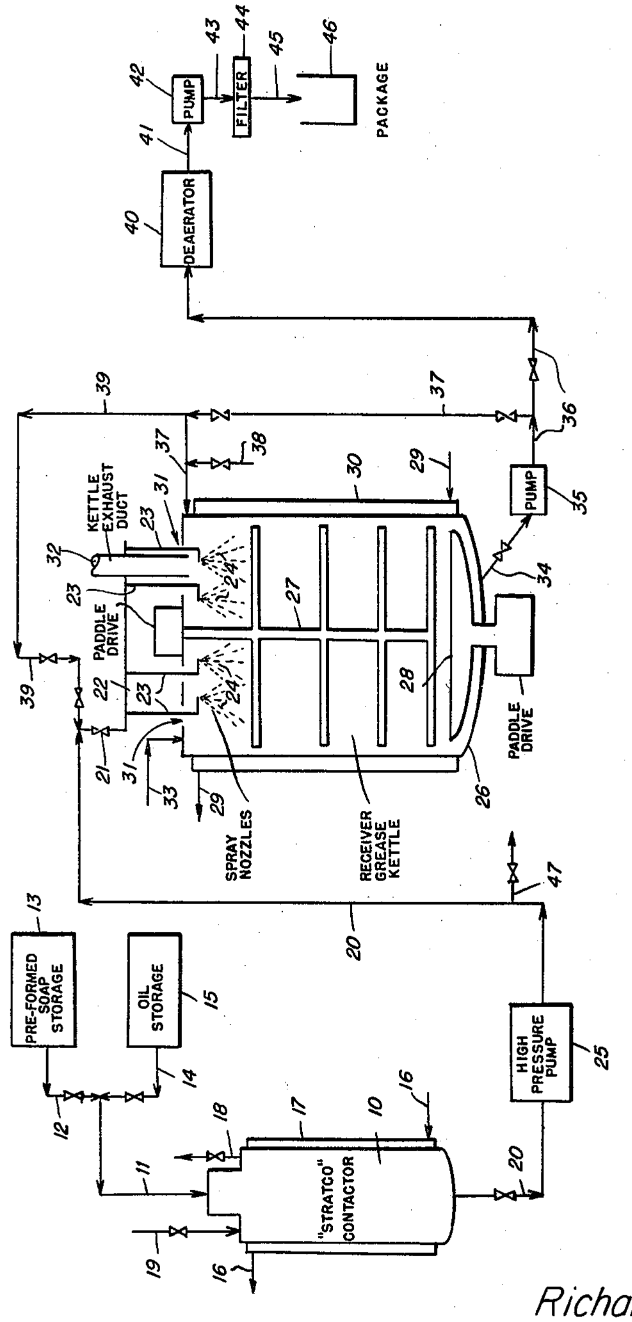

FIG. 1

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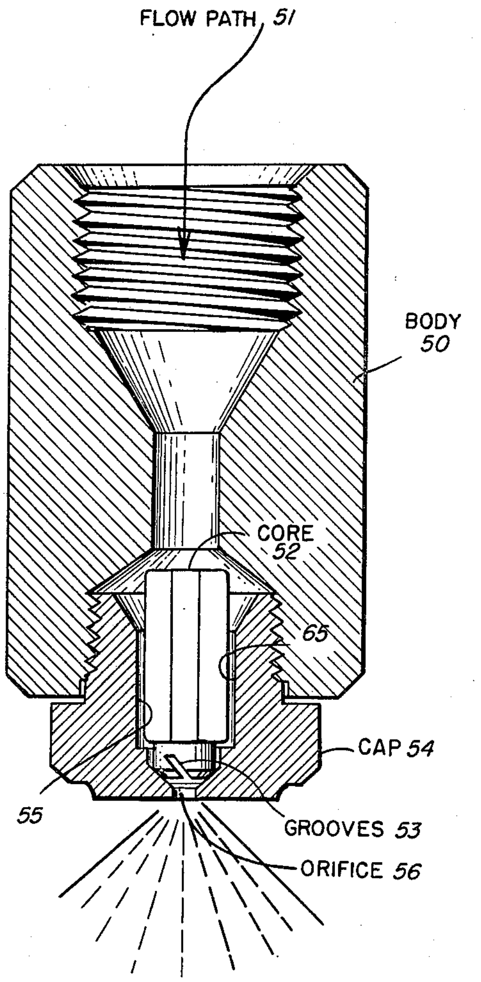
FIG. 2



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GREASE MANUFACTURE

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10 Claims. (Cl. 252—39)

This invention has to do with the art of grease manufacture. More specifically, the invention is concerned with mechanical atomization of a pre-formed soap thickener and a vehicle to produce a grease.

In application Serial No. 682,461, filed September 6, 1957, and issuing August 23, 1960, as Patent No. 2,950,248, Armstrong, Murray and I have described the manufacture of grease with in situ soap formation. Such manufacture involves constituting a mixture of an oleaginous vehicle and soap-forming material. A soap is formed in situ in the vehicle. The resulting vehicle-soap mixture, at a temperature below its solution temperature, is subjected to mechanical atomization into dispersed droplets. The latter are instantaneously contacted directly with a substantially cooler surrounding atmosphere to effect heat exchange thereof. In this way, a grease is formed.

In application Serial No. 683,815, filed September 13, 1957, and issuing August 23, 1960, as Patent No. 2,950,249, Armstrong et al. have revealed a multi-stage grease manufacture in which in situ soap formation is also involved. This comprises: constituting a mixture of an oleaginous vehicle and soap-forming material; forming a soap in situ in the vehicle, at a temperature above, at or below its solution temperature, such that a minor quantity of water is present therewith; subjecting the resulting vehicle-wet soap mixture, at a temperature below its solution temperature, to mechanical atomization to form dispersed droplets and instantaneously contacting the droplets directly with a surrounding atmosphere, whereupon the vehicle-wet soap mixture is substantially dehydrated; and thereafter subjecting the substantially dehydrated product, at a temperature below its solution temperature, to a more intense mechanical atomization to form dispersed droplets, whereupon homogenzation is obtained, and instantaneously contacting the droplets directly with a substantially cooler surrounding atmosphere to effect heat exchange thereof.

As pointed out in said application Serial No. 683,815, "solution temperature" is that temperature at which substantially complete solution of a soap thickening agent in the liquid lubricant occurs. Thus, it is that temperature at which the soap thickening agent is present as discrete molecules or at most molecular aggregates (crystal nuclei) approximately colloidal dimension in size. As a further expression, it is that temperature at which the Tyndall beam disappears in the mixture.

While each grease is characterized by a solution temperature, it is also often characterized by one or more "transition" temperatures. That is, a soap can exist in different crystalline structures while the soap is in the solid state, insoluble in the oil portion. These crystalline structures differ in degree of crystalline order and degree of interpenetration of oleaginous vehicle with the soap crystalline structure. Each crystalline soap structure is stable over a definite but limited temperature range. Thus, the temperature at which a change occurs in the crystalline structure of the soap portion of a grease, is "a transition temperature." It may also be regarded as a temperature at which a phase change occurs in the grease. Such a temperature is less than the already defined solution temperature.

I have now discovered that, in contrast to the foregoing manufactures in which in-situ soap formation is involved, a grease can be formed by mechanically atomizing a mixture of a pre-formed soap or soaps and an oleaginous vehicle. The new process comprises: wetting a preformed soap with a vehicle at a temperature between about 250° F. and the decomposition temperature of either of the soap or vehicle, whichever is lower; subjecting the resulting soap-vehicle mixture at a temperature within the aforesaid range to mechanical atomization under a pressure of at least about 1000 p.s.i. and preferably at least about 2000 p.s.i., to form dispersed droplets; and instantaneously contacting the droplets directly with a substantially cooler surrounding atmosphere to effect heat exchange. The product so formed is a grease.

As used herein, the term "grease" denotes a composition comprising a major proportion of an oil of lubricating viscosity thickened to a grease consistency with a soap or soaps, the composition having an unworked penetration, when tested by ASTM Method D217–52T, of at most 475, using the modified cone recommended by ASTM Technical Committee G, of ASTM Committee D–2.

Accordingly, the primary object of the present invention is to provide a method for readily preparing a grease from a preformed soap or soaps.

Another object is to provide a continuous process in which products of uniform characteristics can be obtained.

Still other objects will be apparent from the following description.

In order that the invention can be readily understood, reference is now directed to the drawings which are attached to and form a part of this specification.

FIGURE 1 is a highly-diagrammatic form of a typical system for practicing the invention.

FIGURE 2 shows a cross-section of a high pressure atomizing nozzle suitable for use herein.

Referring now to FIGURE 1, **10** is a contactor such as for example the "Stratco Contactor," supplied by the Stratford Engineering Company and well known in the art, in which adequate mixing of charge materials accompanies heating. It is to be understood that a pressure kettale, autoclave, etc. can be used in place of a Stracto Contactor, but the latter is preferred. Line **11** is a charge line for introducing soap and oil to contactor **10**. Line **12** carries pre-formed soap from storage **13** and connects with line **11**. Similarly, line **14** carries oil from storage **15** and connects with line **11**.

Heat is supplied to **10** by circulating hot oil, steam or the like through line **16** and the jacket **17** thereof.

In **10** the pre-formed soap and oil are heated, to a temperature and for a time sufficient to ensure wetting of soap with oil. This temperature will be above about 250° F. and below the decomposition temperature of the soap or oil, whichever is lower.

Line **18** is provided at the top of contactor **10**, to serve as a vent, should it be desired to control pressure in the contactor or to allow any volatile material in the charge to escape.

When mixing of ingredients in **10** is complete, air or other gas can be introduced into line **19** under pressure to convey the heated mixture though line **20** and valve **21** to spray manifold **22**, lines **23** and nozzle **24**. The gas pressure should be at least sufficient for the atomization required in nozzles **24**; that is, the pressure should be at least about 1000 p.s.i. Spray manifold **22**, for example, can comprise a 2 inch pipe having four ½ inch pipes **22**. Each of lines **22** is equipped with an atomizing nozzle **24**.

In line **20**, there may also be high pressure pump **25** to effect transfer of the heated mixture in contactor **10** to valve **21** and spray manifold **22**.

The mixture in lines **23** is a mixture of soap wetted with oil. The mixture does not have the consistency of a grease, nor does the soap separate from the oil. The mixture in lines **23** passes through nozzles **24** into receiver **26** such as a grease kettle equipped with either or both of a motor-driven, multi-bladed agitator **27** and a motor-driven, paddle-type agitator **28**. These agitators serve to effect removal of product from the walls and bottom of kettle **26**. As shown, the temperature of product in **26** can be regulated by oil, steam or the like circulating through line **29** and jacket **30**. Finely dispersed droplets are discharged from nozzles **24** into receiver **26** and are contacted with a surrounding atmosphere in receiver **26**. As the product emerges from nozzles **24**, it is cooled substantially by the surrounding atmosphere. Air can be admitted to receiver **26** through inlets **31** surrounding lines **23**, to regulate the amount of cooling desired of the product discharged from nozzles **24**. It is to be understood that atmospheres other than air can be used and that such can be introduced through inlets **31**. For example nitrogen, carbon dioxide, flue gas, steam and the like can be used.

Should any water be present in the soap-vehicle mixture charged to nozzles **24**, it is removed substantially as water vapor at the discharge side of the nozzles. Water so flashed from the product escapes through duct **32**. Since part of this water may condense to droplets of liquid water, air admitted through inlets **31** sweeps out through **32** a mixture of water vapor and water droplets. However, it has been found that water need not be present in the charge. In fact, no advantage is gained when water is so present.

In the event a grease concentrate is obtained in the discharge from nozzles **24** by using only part of the entire oleaginous vehicle, instead of a mixture with all of the vehicle, all or part of the remainder of the vehicle or vehicles can be added to receiver **26** through line **33**. It is to be understood that one or more additives can also be charged, in part or in entirety, through line **33**.

Grease in receiver **26** is removed through valved line **34** by pump **35** and is discharged into valved line **36**. If desired, the product can be recycled through valved line **37** for return to receiver **26**. This is advantageous to aid in mixing vehicle or additive charged through valved line **38** with product collected in receiver **26**. The product in line **36** can also be recycled through lines **37** and **39** to nozzles **24** by way of **21**, **22** and **23**. This is advantageous in the event further atomization of the grease product should be desired.

The grease product in receiver is generally highly aerated. It is removed through valved line **34** by pump **35** and is passed through line **36** to deaeration in deaerator **40**. The latter can be any of those usual in the art, such as a Morehouse Deaerator, a Cornell Cold Grease Homogenizer, a Kinney Heli-Quad vacuum pump, or of the type described by Brooke and Piazza in U.S. Patent No. 2,797,767. These devices generally operate on a vacuum principle. Grease emergent from deaerator **40** through line **41** can be pumped by pump **42** and line **43** through a conventional filter **44**. The finished grease is taken through line **45** and is packaged in equipment designated **46**.

Line **47**, in line **20**, is provided as a pressure release line for safety purposes.

FIGURE 2 reveals, in cross-section, a typical high pressure atomizing nozzle found effective for the mechanical atomization of this invention. This is composed of body **50** containing flow path **51**. Toward the end of flow path **51** there is a removable core **52**, of hexagonal or square cross-section, and having grooves **53**. The core is held in place by orifice cap **54** which is secured to body **50** as shown. As material flows through path **51**, it passes along core **52** through annular passage **55** defined by core **52** and cap **54**. Material is expelled through orifice **56** of cap **54**. The material acquires tangential velocity components in passing through the grooves **53** in core **52**. This causes the stream of material to exit from orifice **56** as a hollow cone which atomizes into a hollow-cone spray. This fitting of FIGURE 2 is of heavy construction, being designed for pressures in passage **55** of at least 1000 p.s.i. and preferably up to several thousands of pounds per square inch. The dimensions of one grooved-core nozzle found useful in the examples provided hereinbelow, were: orifice diameter, 0.134 inch; a six-grooved core, each groove cross-section being 0.050 by 0.065 inch. Such a nozzle is supplied by Spraying Systems Company, and is identified as ½ SB 30 Nozzle, Number 40 Core.

In place of the atomizing device illustrated by FIGURE 2, other such devices known in the art can be used so long as the operating pressure is at least about 1000 p.s.i. For example, the following can be mentioned: impinging jet nozzles, centrifugal or rotating disc atomizers, pneumatic atomizers, vibrating atomizers, multi-jet atomizers, impact type nozzles and other liquid dispensing devices.

Typical examples are provided in order to illustrate various facets of the invention.

EXAMPLE 1

This involves the preparation of a grease of the character defined in U.S. Patent No. 2,842,494. The grease is comprised of lithium stearate (8 percent by weight), lithium soaps of wool grease fatty acids (3 percent by weight), dipropylene glycol pelargonate (66.5 percent by weight) and a naphthenic mineral oil, 750 seconds S.U.S. at 100° F. (22.5 percent by weight). The grease has a solution temperature of 370° F.

The oils and soaps were charged to contactor **10**, 100 pounds size, and were mixed and heated to 420° F. over a period of 2 hours. The piping arrangement **20**, **21**, **22** and **23** was preheated to about 420° F. The contactor was pressured with air from line **19** such that the soap-oil mixture was forced through the piping arrangement to a single high-pressure nozzle **24**. The temperature of the mixture charged to the nozzle was 415–412° F. As the product was expressed from the nozzle, its temperature was about 220° F. The nozzle pressure was between 2200 and 2700 p.s.i. The product was sprayed into and collected in a drum receiver **26** and passed to a Cornell Cold Grease Homogenizer to remove air. Characteristics of the grease so obtained are shown in Table I following wherein it is identified as "Grease Example No. 1." Several related products are shown in the same table.

Table I

Grease Example No	1	2	3A	4A
Total soap content, Wt. percent	11	11	11	11
(1) Li WGFA, Wt. percent	3	3	8	8
(2) Lithium stearate, Wt. percent	8	8	3	3
Ratio (1)/(2), Wt. percent	0.38/1	0.38/1	2.7/1	2.7/1
Solution temperature, ° F	370	370	370	370
Contactor temperature, ° F	415-412	350-342	450	348-352
Nozzle inlet temperaature, ° F	376-384	350-342	380	330
Nozzle pressure, p.s.i	2,200-2,700	2,600-3,500	3,100	2,900-3,000
Penetration (ASTM):				
Unworked	275	230	245	197
Worked 60×	312	280	272	230
Worked, 50,000 strokes, 1/16″ Holes	337	359	306	296
Roll stability—2 hours:				
Micro penetration, Initial	90	69	66	45
Micro penetration, Final	122	137	112	77

A—charge contained 89 weight percent of ester vehicle.

EXAMPLE 5

This involves the preparation of a lithium soap grease composed of lithium 12-hydroxy stearate (8 pounds; 8 percent by weight) and a naphthenic oil, 750 seconds

S.U.S. at 100° F., (92 pounds; 92 weight percent). The grease has a solution temperature of 383° F.

The oil and soap were charged to contactor 10, 100 pounds size, and were mixed and heated to 375° F. over a period of 1½ hours. The piping arrangement 20, 21, 22 and 23 was preheated to about 375° F. The contactor was pressured with air from line 19 such that the soap-oil mixture was forced to high pressure pump 25. From the pump 25, the mixture passed through 21, 22 and 23 to a single high-pressure nozzle 24. The temperature of the mixture charged to the nozzle was from 365–358° F. As the product was expressed from the nozzle, its temperature was about 200° F. The nozzle pressure was between 3100 and 3600 p.s.i. The product was sprayed into and collected in a drum receiver which served as receiver 26. The grease so obtained was smooth in texture and had the following characteristics.

Penetration (ASTM):	
Unworked	325
Worked 60×	338
Worked, 50,000 strokes, 1/16" holes	399
Roll stability—2 hours:	
Micro penetration, initial	125
Micro penetration, final	187

A further illustration of the preparation of a grease of the type shown in Example 5 is identified in the following tabulation as Example 6.

EXAMPLE 6

Contactor temperature, ° F	418–414
Nozzle inlet temperature, ° F	400–395
Solution temperature, ° F	383
Nozzle pressure, p.s.i	2700–3000
Penetration (ASTM):	
Unworked	251
Worked 60×	263
Worked, 50,000 strokes, 1/16" holes	339
Roll stability—2 hours:	
Micro penetration, initial	80
Micro penetration, final	119

EXAMPLE 7

A lithium soap grease was prepared from the following materials:

Lithium stearate	pounds	1.35
Lithium 12-hydroxy stearate	do	5.40
Lithium hydroxide monohydrate	grams	15
Water	do	45
Naphthenic mineral oil (see Example 1)	pounds	93

Lithium hydroxide monohydrate was included in the charge to ensure neutralization. Water was included to solubilize the hydroxide.

Soaps and oil at 250° F. were taken from storage 13 and 15, and were charged to contactor 10. Lithium hydroxide monohydrate and water were heated to 212° F. and charged to the contactor. The latter was closed, and the materials were mixed and heated therein to 372° F. over a period of 1½ hours. The contactor was then vented by opening valved line 18. The mixture in the contactor was removed through line 20 as described in Example 5. The entire pipe system 20 through 23 was heated to about 350° F.

The temperature of the mixture charged to a single high-pressure nozzle was 355° F. Nozzle pressure was 3000–4500 p.s.i. After the product was collected, it was passed through a Cornell Cold Grease Homogenizer. The final product was a firm, transparent grease of the following nature.

Penetration (ASTM):	
Unworked	267
Worked	275
Worked, 50,000 double strokes, 1/16" holes	367
Roll stability—2 hours:	
Micro penetration, initial	87
Micro penetration, final	144

EXAMPLE 8

A lithium grease was prepared from 7.5 pounds of lithium stearate and 42.5 pounds of dipropylene glycol dipelargonate. The solution temperature of this grease is 370° F. The procedure used was that described in Example 5, operating conditions and characteristics of the product being shown below:

Contactor temp., ° F	390–382
Nozzle inlet temp., ° F	380–377
Nozzle pressure, p.s.i	2400–2700
Penetration (ASTM):	
Unworked	274
Worked	280

The penetration values reported are those obtained on the collected product after it has been passed through a Cornell homogenizer.

EXAMPLE 9

This example is given to illustrate a lithium grease composed of lithium stearate and lithium-12 hydroxystearate. The vehicle used was dipropylene glycol dipelargonate.

Total soap content, wt. percent	10
(1) Lithium 12-hydroxystearate, wt. percent	8
(2) Lithium stearate, wt. percent	2
Ratio (1)/(2), wt. percent	4/1
Solution temperature, ° F	383
Contactor temperature, ° F	423–425
Nozzle inlet temperature, ° F	377–384
Nozzle discharge temperature, ° F	200
Nozzle pressure, p.s.i	2500–2700
Penetration (ASTM):	
Unworked	252
Worked 60×	272
Worked, 50,000 strokes, 1/16" holes	354
Roll stability—2 hours:	
Micro penetration, initial	75
Micro penetration, final	109

EXAMPLE 10

Illustrated here is a lithium stearate grease. Fifteen percent by weight of such soap was used with 85 percent by weight of dipropylene glycol dipelargonate. Operating conditions and characteristics of the product are given below:

Contactor temperature, ° F	390–382
Nozzle inlet temperature, ° F	380–377
Nozzle pressure, p.s.i	2400–2700
Penetration (ASTM):	
Unworked	274
Worked	280

EXAMPLE 11

Provided here is an illustration of a mixed base grease comprised of lithium and calcium stearates. The grease was formed from lithium stearate (7 percent by weight), calcium stearate (2 percent by weight) and a naphthenic mineral oil having a viscosity index of 10. The grease has a solution temperature of 366° F. The procedure used was that which is described above in Example 6.

Contactor temperature, ° F	356
Nozzle inlet temperature, ° F	346
Nozzle pressure, p.s.i	3300–3700
Penetration (ASTM):	
Unworked	260
Worked, 60×	274
Worked, 50,000 strokes, 1/16" holes	374
Roll stability—2 hours:	
Micro penetration, initial	82
Micro penetration, final	140

EXAMPLE 12

This example shows calcium soap greases in an ester vehicle, dipropylene glycol dipelargonate. Calcium acetate monohydrate, calcium caprylate and calcium stearate in the percent weight balance respectively of 11.6, 8.0 and 3.7, were employed. The salt-soap content, therefore was 23.3 percent by weight. The balance was ester vehicle.

In one illustration (A), the mixture of salts, soap and vehicle was heated slowly, with constant mixing, to 450° F. The mixture was then cooled to about 300° F. and passed through a high pressure nozzle. In a second illustration (B), the mixture was heated to 400° F. and then atomized. A summary of operating conditions and of the products is given below:

	A	B
Contactor Temperature, ° F	450	400
Nozzle Inlet Temperature, ° F	275–285	375
Nozzle Pressure, p.s.i	1,000–2,500	2,000
Penetration (ASTM):		
Unworked	265	253
Worked 60×	293	273
Worked, 50,000 strokes, 1/16″ holes	307	389
Roll Stability—2 Hours:		
Micro Penetration, Initial	71	78
Micro Penetration, Final	133	102
Timken O.K. Load, r.p.m., pounds	—	15

Considering the data given above, several features merit attention. With lithium greases wherein a substantial part or all of the soap is derived from a hydroxy acid, the atomization temperature should be above, at or only slightly below the solution temperature. When the atomization temperature is substantially below the solution temperature, greases of softer consistency are formed. This obtains whether a mineral oil or synthetic ester vehicle is used. Therefore, in the preparation of such lithium soap greases it is preferable that the atomization temperature be not more than about 20° F. below the solution temperature. In contrast, greases containing a mixture of lithium stearate and lithium wool grease fatty acid soaps prepared with an atomization temperature below the solution temperature are firmer than corresponding greases prepared with an atomization temperature above the solution temperature.

Oleaginous vehicles useful herein have been illustrated above by mineral oils and synthetic esters. It is to be understood, however, that other oils of lubricating viscosity can also be used in the present invention. Typical of such vechiles are polymerized olefins, silicones, fluorocarbons, perfluoroalkyl ethers, esters of polybasic acids, esters of polyalcohols and monocarboxylic acids, silicate esters, esters of phosphorus-containing acids, amines, etc. Illustrative of such vehicles are: polypropylene, polypropylene glycol, di-(2-ethyl hexyl) sebacate, di-(2-ethyl hexyl) adipate, dibutyl phthalate, polyethylene glycol di-(2-ethyl hexoate), polymethylsiloxane. The synthetic

vehicles are most suitable for providing greases for use in aircraft, since many of these greases retain their lubricating value over a wide temperature range, from about −100° F. to about 500° F. In general, the mineral oils and synthetic lubricants which can be used herein are characterized by a viscosity (S.U.V.) of greater than about 40 seconds at 100° F., preferably from about 60 to about 6000 seconds at 100° F.

Although the invention has been illustrated above by soaps of lithium, of lithium and calcium, and of calcium alone, it is to be understood that soaps of any metals useful in forming grease compositions can be used in the present invention. For example, soaps of one or more metals of the following group can be used: sodium, potassium, lithium, calcium, barium, strontium, zinc, aluminium, etc.

What is claimed is:

1. The method of making a grease which comprises: wetting a pre-formed soap thickening agent in a grease-forming quantity with an oil of lubricating viscosity, at a temperature between about 250° F. and the lower of the decomposition temperatures of the said agent and oil; subjecting the resulting oil-soap mixture at a temperature within the aforesaid range and at a pressure above about 1000 pounds per square inch, to mechanical atomization into dispersed droplets, and instantaneously contacting the droplets directly with a substantially cooler surrounding atmosphere to effect heat exchange thereof, thereby forming a grease.

2. The method of claim 1 wherein the oil is a mineral oil.

3. The method of claim 1 wherein the oil is a synthetic ester.

4. The method of claim 1 wherein the oil is dipropylene glycol dipelargonate.

5. The method of claim 1 wherein the soap thickening agent is a lithium soap.

6. The method of claim 1 wherein the soap thickening agent is a mixture of lithium soaps.

7. The method of claim 1 wherein the soap thickening agent is a mixture of calcium salts and soaps.

8. The method of claim 1 wherein the soap thickening agent is a complex of calcium salts and soaps.

9. The method of claim 1 wherein the pressure is above about 2000 p.s.i.

10. The method of claim 1 wherein the soap thickening agent contains a substantial proportion of a lithium hydroxyacid soap and the oil-soap mixture is at a temperature no more than about 20° F. below its solution temperature when subjected to said atomization.

References Cited in the file of this patent

UNITED STATES PATENTS

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2,950,248	Armstrong et al.	Aug. 23, 1960
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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,108,965 October 29, 1963

Richard A. Butcosk

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 59, for "though" read -- through --; column 7, in the table, last line thereof, for "Timken O.K. Load, r.p.m." read -- Timken O.K. Load, 800 r.p.m. --; same column 7, line 51, for "vechiles" read -- vehicles --.

Signed and sealed this 30th day of June 1964.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents